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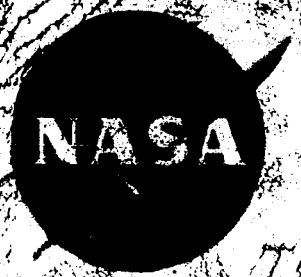

RESEARCH PROGRAM FOR THE LONG TERM TESTING OF CYLINDRICAL DIODES AND THE IRRADIATION OF FUEL AND INSULATORS

Seventh Quarterly Progress Report
December 18, 1964 - March 17, 1965

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-2544

GENERAL ELECTRIC COMPANY
Special Purpose Nuclear Systems Operation

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SEVENTH QUARTERLY SUMMARY REPORT

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April 18, 1965

CONTRACT NAS 3-2544

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RESEARCH PROGRAM FOR THE LONG TERM TESTING OF CYLINDRICAL DIODES AND THE IRRADIATION OF FUEL AND INSULATOR

ABSTRACT

The experimental and analytical work in nuclear thermionics being performed by the Special Purpose Nuclear Systems Operation of the General Electric Company for the National Aeronautics and Space Administration under Contract NAS 3-2544, "Research Program for the Long Term Testing of Cylindrical Diodes and the Irradiation of Fuel and Insulator," is presented. Information from the literature, information generated under the contract, and information generated by the contractor in other studies, some of which were conducted under other contracts with the government, are included. The work is administered under the direction of the Nuclear Power Technology Branch, Lewis Research Center, Cleveland, Ohio.

RESEARCH PROGRAM FOR THE LONG TERM TESTING OF CYLINDRICAL DIODES AND THE IRRADIATION OF FUEL AND INSULATOR

SECTION I

SUMMARY

This is the Seventh Quarterly Summary Report describing progress on the research program for the long term testing of cylindrical diodes and the irradiation of fuel and insulator under Contract NAS 3-2544. The program consists of work phases; (1) development of basic converter life, (2) thermal and irradiation testing of fuel/clad emitter, and (3) irradiation of high purity alumina at temperature.

The objectives and scope of each of these work areas are as follows:
Phase A: Develop, life test, and evaluate basic thermionic converter designs.

Task 1: Design, fabricate, and life test three cylindrical converters to demonstrate the life of collector, emitter, and spacing assemblies and related internal components. Dissect components to investigate causes of deterioration or failure.

Task2: Design, fabricate, and life test three converters similar to those in Task 1 which includes a mockup of an intercell assembly for multicell application. This intercell assembly includes provision for electrode spacing, back emission shields, and intercell connections. Intercell connections are not required to carry current. Dissect components to investigate causes of deterioration or failure.

Task 3: Perform detail design (including thermal and stress analyses) and prepare procedures for fabrication and assembly for a three converter series connected segment of a thermionic fuel element.

Task 4: Design, fabricate, and life test two thermionic converters which include a mockup of an intercell assembly based on the physical design of Task 3. The converter tests are to be conducted at an emitter temperature of 1800°C and a collector temperature of 1000°C for 2000 hours or until failure, whichever occurs first. The intercell electrical connections shall be active, i.e. current conducting. Dissect converters and investigate causes of component failure and deterioration.

Task 5: Design, fabricate, and life test three converters to investigate the effect of a UO_2 fuel in a W-25 w/o Re clad emitter on thermionic performance during an extended period (4500 hours) of converter operation at an emitter temperature of 1800°C and a collector temperature of 1000°C. Dissect converter and investigate failures or physical changes noted.

Phase B: Develop, fabricate, and study the long-term effects on Fuel/clad emitters from thermal and irradiation exposures.

Task 1: Design, fabricate, and test twelve W-25 w/o Re UO_2 fueled specimens in high temperature furnaces in the temperature range 1800°C to 2200°C for periods up to 5000 hours in a cesium vapor environment. Subject a representative number of the specimens to detailed analyses to determine the nature and extent of any fuel/clad interactions, and the effects of venting the fuel to the cesium environment.

Task 2: Fabricate and irradiate two irradiation capsule assemblies containing specimens of uranium oxide fuel clad in vented tungsten 26% rhenium in Plum Brook Reactor Facility. Perform post-test analyses to determine the dimensional stability structural integrity, and extent of any interaction for the irradiated specimens.

Phase C: Irradiation of high purity alumina at temperature. Expose alumina specimens to irradiation in the Plum Brook Reactor in a vacuum environment. Measure electrical resistivity and breakdown voltage during irradiation and perform post-irradiation examination to determine changed in physical properties of the specimens.

A summary of the work performed during the seventh quarter and contained in Section III of this report follows:

Phase A: Develop, fabricate, and evaluate Thermionic Converter Designs

Task 2: Out-of-pile converters 311, 312, and 313 were operated for 6413, 5807, and 3775 hours respective. Converter 311 failed at 6413 hours by indicating cesium leakage into the bombardment chamber. Converter 313 failed similarly after having been thermal cycled 6 times. Converter 312 is continuing in test.

Task 3: a) The stress analysis and design of the TFE were completed and forwarded to the NASA Project Manager. The out-of-pile Converter design was initiated.

Task 5: Extensive fabrication of the W-25% Re fueled converter was done. Two filaments were calibrated and the first emitter assembly was made for emitter calibration.

Phase B: Thermal and irradiation testing of fuel/clad emitters.

Task 1: Long term high temperature compatibility tests of UO_2 clad with W-25% Re.

Thermal exposure of capsules 1, 2, 5, 6, 7, 8, 9, 10, is being continued. Exposure of capsules 3, 4, 11, and 12 has been concluded, and the capsules have been examined. Time and temperature for exposure of these four capsules are as follows:

Capsule	Temperature	Time
3	1800°C	500 Hours
4	1800°C	1000 Hours
11	2200°C	500 Hours
12	2200°C	1000 Hours

No significant changes in dimension were observed for any of the capsules. No significant weight changes were determined for capsules 3 and 4, but capsules 11 and 12 showed considerable weight loss because of the rupture of the cesium containment during the first 500 hours.

No gross interaction between UO_2 and W-25% Re was disclosed by metallographic examination.

Task 2: Fuel Clad Irradiation

The experiment has been redesigned to attain a burnup in the UO_2 fuel of 1×10^{20} fissions/cm^3 with a minimum irradiation time, and utilizing as many of the fabricated capsule components as possible. The Design and Hazards Manual has been modified to reflect the redesign of the experiment. The Vertical Adjustable Facility Tube (VAFT) has been completed and operational checks and accuracy and repeatability tests have been completed with satisfactory results.

Phase C: Irradiation of Alumina

Analysis were performed by the PBR to more accurately define the fast flux history of capsule irradiations reported in the Sixth Quarterly Report.

The vacuum can containing the samples was removed from the irradiation capsule and shipped from the Plum Brook Reactor Facility to the Vallecitos Atomic Laboratory for post irradiation examination.

SECTION II

INTRODUCTION

To meet future space power requirements over the range of kilowatts to megawatts with a light-weight, reliable and long-life system, the Special Purpose Nuclear Systems Operation of General Electric has proposed the STAR-C (Space Thermionic Auxiliary Reactor).

A significant feature of the STAR-C is the integral thermionic fuel element which consists of a number of nuclear-fueled series-connected, power-converting diodes. Emphasis has been placed on this development.

The attainment of the high reliability levels which are required for a space power system will require each individual, series-connected thermionic cell to be fabricated and perform to exacting reliability standards. Development work on materials and converters to achieve the goals of reliability and life in configurations acceptable to the thermionic reactor system is supported as part of the present program as well as under other nuclear thermionic programs at the General Electric Company.

This is the Seventh Quarterly Report describing progress on the research program for the long term testing of cylindrical diodes and the radiation of fuel and insulators under Contract NAS 3-2544. The program consists of three work areas:

A. Development of Basic Converter Life

B. Thermal and Irradiation Testing of Fuel/Clad Emitter

C. Irradiation of High Purity Alumina at Temperature

Objectives of each of these and technical progress during the quarter are summarized in Section III.

SECTION III
TECHNICAL PROGRAM STATUS

A. DEVELOPMENT OF BASIC CONVERTER LIFE

1. General

This phase consists of four converter investigations described as follows:

a. Task 1, Converters 301 - 303

These converters consists of cylindrical tungsten emitters, niobium collectors, and have an interelectrode spacing of 0.01 inch. The detailed design of these converters is described in reference 1. The objective of this task is to develop thermionic converters of high reliability under simulated thermionic fuel element conditions and obtain information on their possible failure modes.

b. Task 2, Converters 311 - 313

These converters consist of cylindrical tungsten emitters, niobium collectors, and have a 0.01 inch spacing with a spacing assembly mounted on one end of the emitter that closely simulated an intercell structure for a thermionic fuel element. The detailed design of this assembly is described in reference 2. The objective of this task is to subject the collector-to-emitter spacing components and intercell assembly to realistic operating conditions and obtain information on their possible failure modes.

c. Tasks 3 and 4, Converters 331 - 332

This sub-task consists of two converters very similar to those in Task 2, the sole exception is that the intercell assembly mounted on one end of the emitter contains the current carrying emitter lead. The converter design is based upon a thermionic fuel element design, including thermal and stress analysis, which is part of the converter design task. The objective of this task is identical to Phase II with the added simulation of the current conduction in the connector pins.

d. Task 5, Converters 431 - 433

These three converters consist of electrically heated cylindrical tungsten 25% rhenium emitters containing uranium dioxide fuel, niobium collectors; and have an interelectrode spacing of 0.01 inch. The objective of this task is to investigate the possible influence of UO_2 on the performance of thermionic converters which employ W-25% Re clad, UO_2 fueled emitters.

2. Task 1, Converters - Series 301 - 303

Testing of these converters had been terminated previously and results of post-test examination of 302 and 303 reported. No post-test examinations had been initated on converter 301 at the end of the reporting period.

3. Task 2, Converters - Series 311 - 313

Converter 311 - From about the 4300-hour point, the adjustable transformer in the bomber voltage supply became mechanically frozen preventing the standard test practice of maintaining the input power constant. In spite of this condition, an indicated increase in effieiency with time was reflected in the output power remaining near 100% while the input power was slowly

decreasing. Near the 6000 hour point, power input had decreased to just above 90% and the emitter temperature had decreased to 1750°C. At 6016 hours, the tests console was turned off and a new bomber supply adjustable transformer was installed. The converter was then placed back in test. 90% of the initial power output was obtained with the input at approximately 90% of the initial value to re-establish the performance just prior to the repair. The emitter temperature was approximately 1750°C and the collector temperature was 700°C. At 6250 hours the converter was temporarily shut down again for an adjustment to the test console. Upon placing the converter back on life test, 110% of the initial output power was obtained with 102% of the initial input power. The emitter temperature was 1815°C and the collector temperature was 709°C. By 6369 hours, the filament had begun to show a fluctuation in the current-voltage relationship. Current-voltage curves were obtained which indicated evidence of cesium in the bombardment chamber. Converter heater failure was declared and the converter tests halted at 6413 hours. No post-test examinations had been initiated at the end of the reporting period. Input and output for converter 311 plotted as functions of time are shown in Figure 1.

Converter 312 - At the end of the reporting period, a total of 5807 hours had been accumulated. The converter was operating at an emitter temperature of 1830°C and a collector temperature of approximately 725°C. Input and output power for converter 312 plotted as functions of time are shown in Figure 2. Individual data points are not shown for the time period 4025 hours to 5807 hours because the input and output powers were varied over a wide range during that period as described in the following paragraphs.

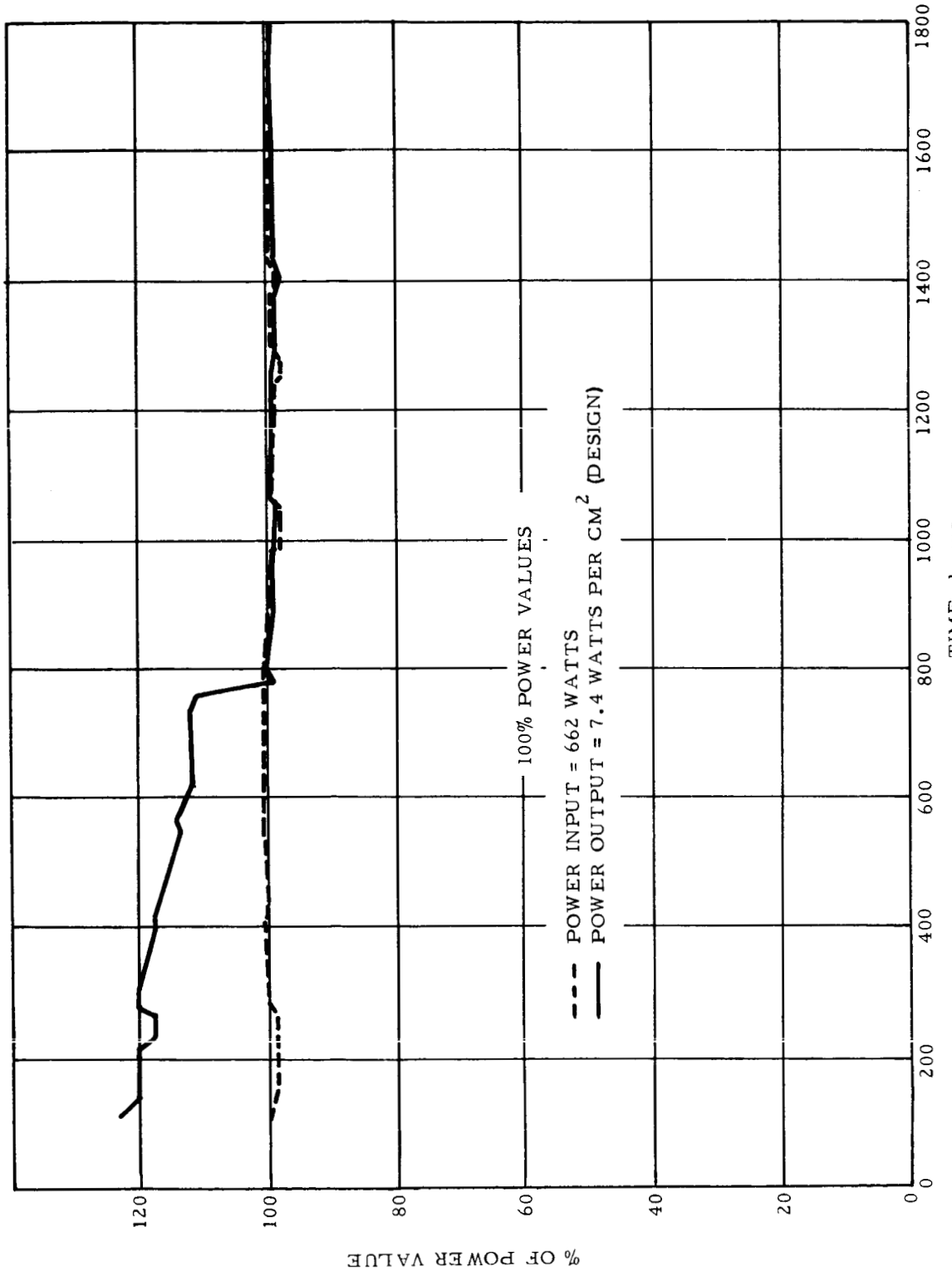


FIGURE 1a CONVERTER 311 POWER INPUT AND OUTPUT vs TIME

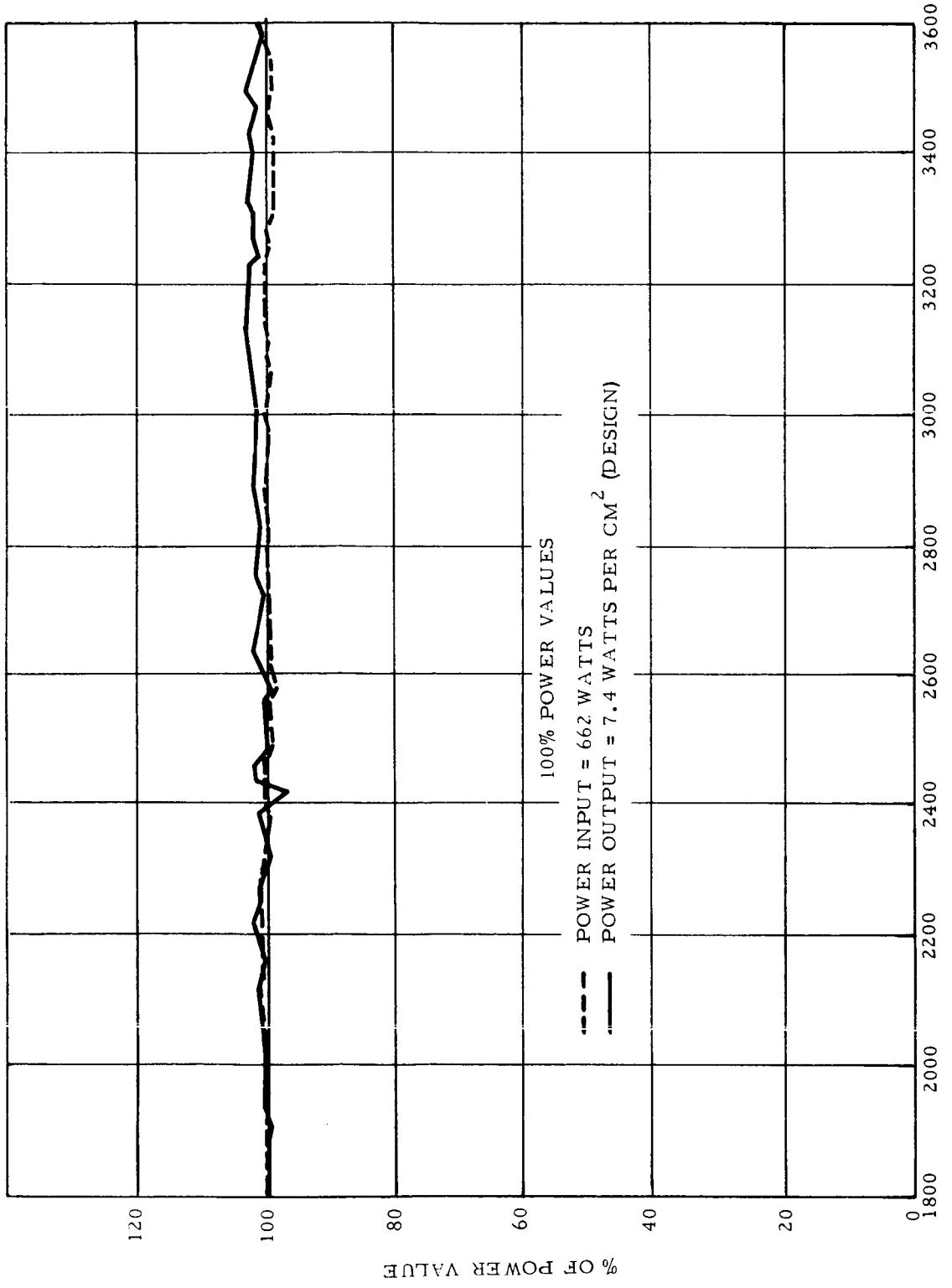


FIGURE 1b CONVERTER 311 POWER INPUT AND OUTPUT vs TIME

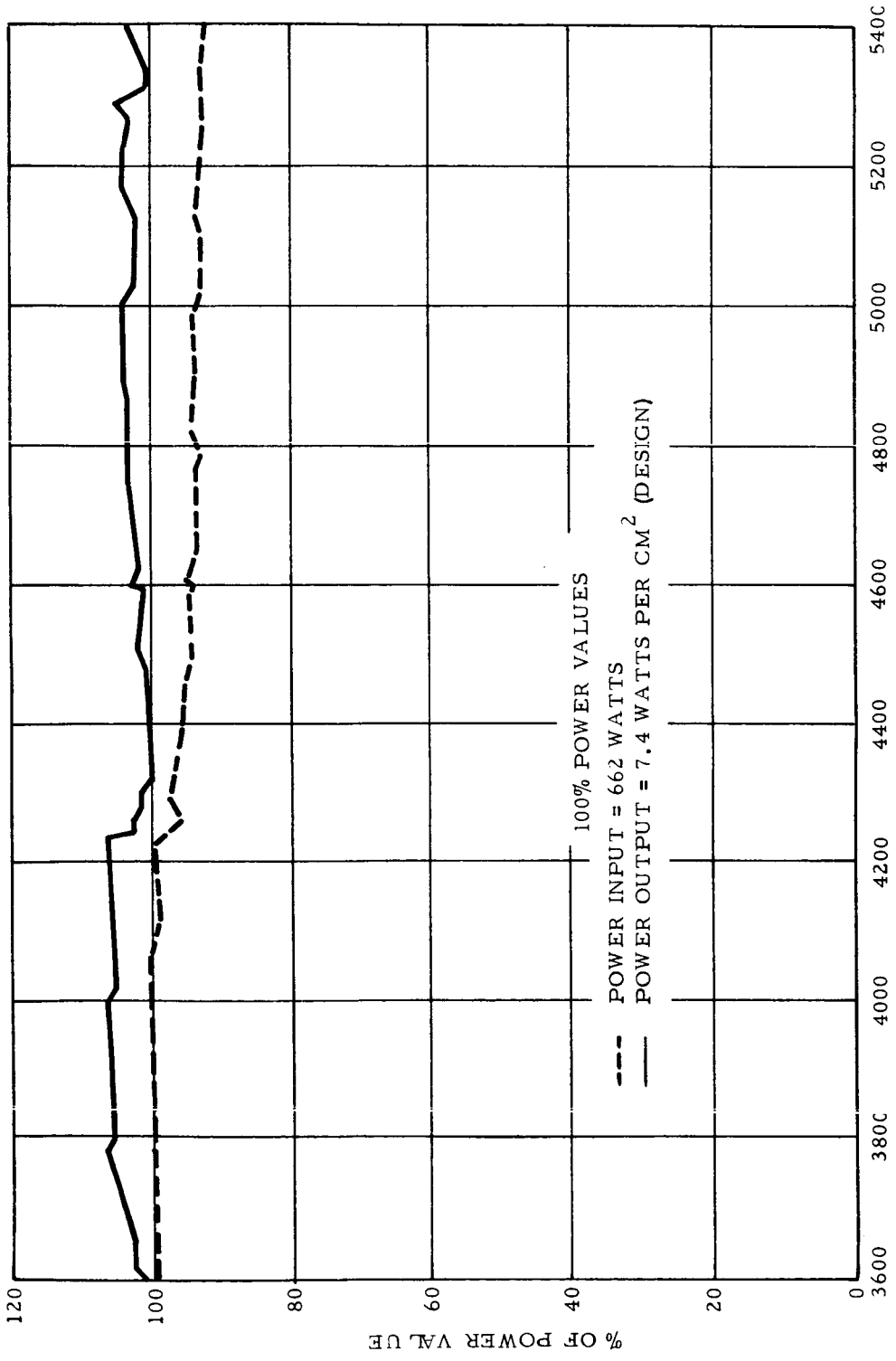


Figure 1c Converter 311 Power Input and Output vs Time

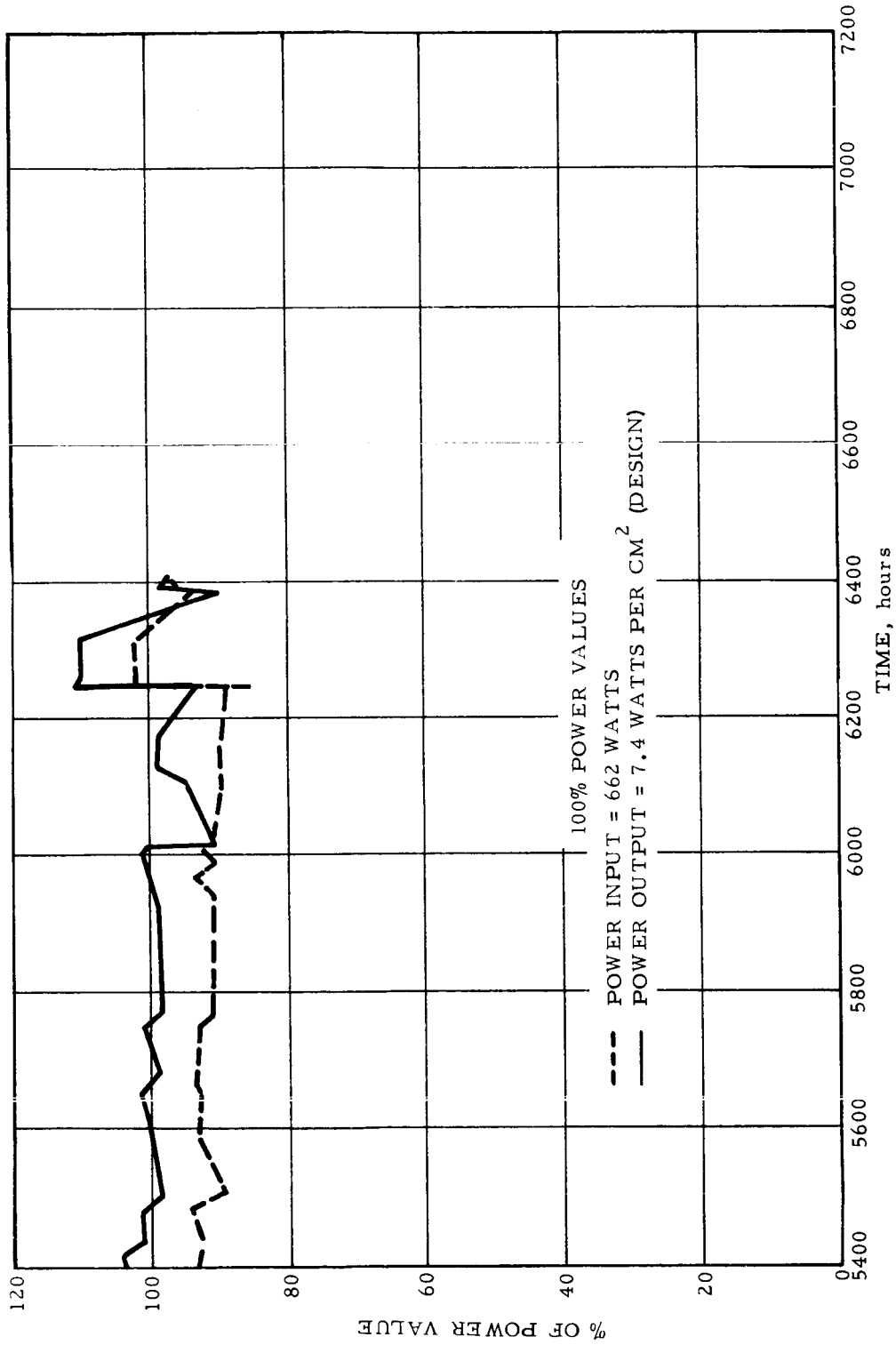


Figure 1d Converter 311 Power Input and Output vs Time

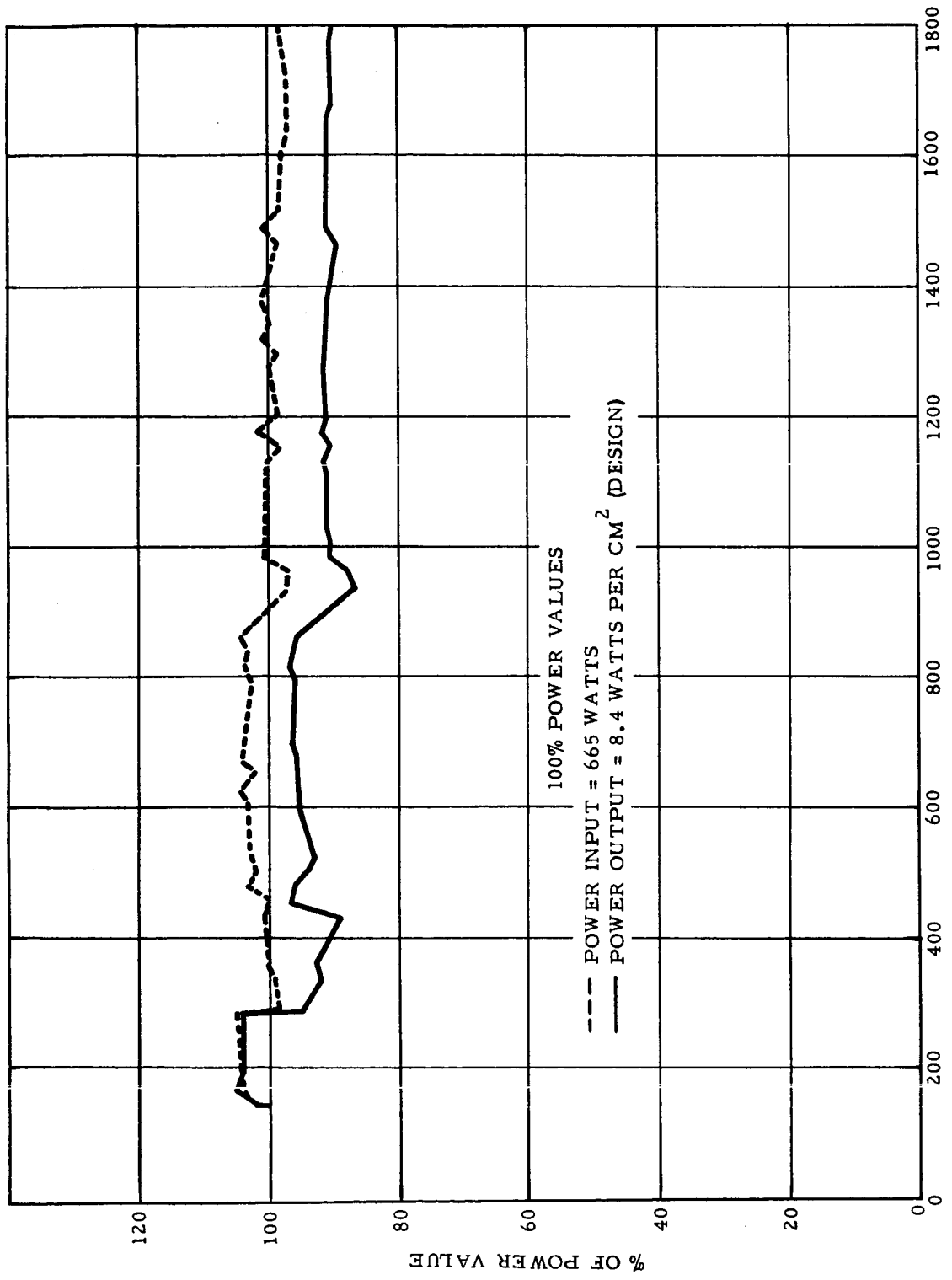


FIGURE 2a CONVERTER 312 POWER INPUT AND OUTPUT vs TIME

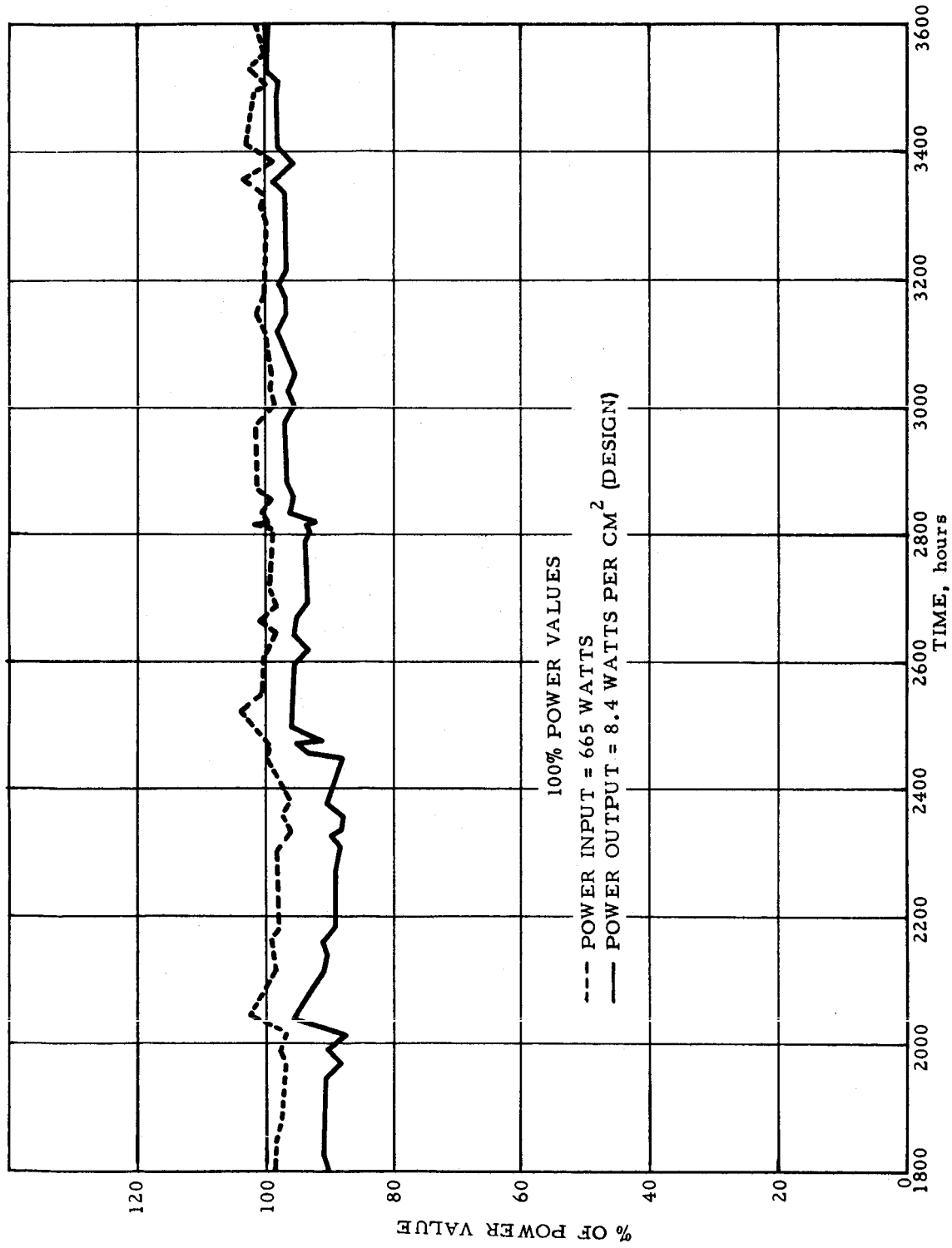


FIGURE 2b CONVERTER 312 POWER INPUT AND OUTPUT vs TIME

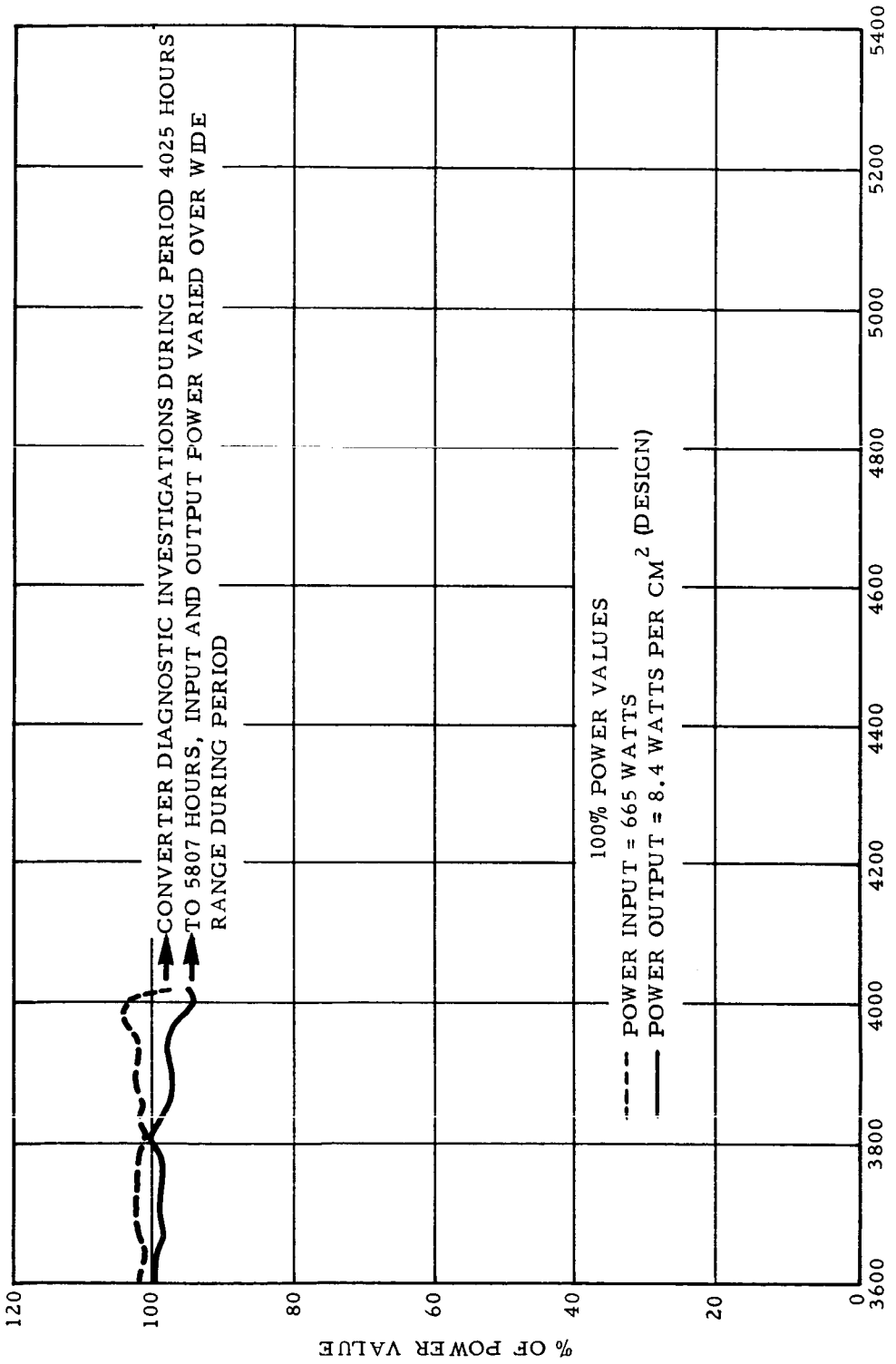


Figure 2c Converter 312 Power Input and Output vs Time

During the reporting period, an investigation was conducted to ascertain the causes, if possible, of an apparent secondary cesium reservoir. This manifests itself by the power output being independent of the cesium reservoir temperature about the 345°C. Below that point, reductions in cesium reservoir temperature cause a repeatable reduction in output power. This was observed with a collector temperature of 700°C. When the collector temperature was revised to 750°C, power output was controlled by the cesium reservoir temperature in a repeatable manner as if the secondary reservoir had disappeared due to the higher collector temperature. Power output of the converter returned to the original value at the corresponding collector temperature; however, a slow loss in power output occurred a few hours thereafter.

Work function measurements on both the emitter and the collector were made at this point. The current vs temperature measurements on the emitter indicate a normal S curve on the Richardson plot. Collector work function measurements indicated a collector work function of about 1.6 volts in the fully cesiated condition. The emitter work function was approaching 4.6 volts at an emitter temperature of 1834°C and a cesium reservoir temperature of 150°C. When the converter was returned to test and the collector temperature raised to 730°C, the power output returned to its original value and remained there for a few hours. Subsequently the power output drifted down as if the cesium reservoir pressure were too low, thus repeating its characteristics of the apparent secondary cesium reservoir.

A systematic re-run of the voltage-current characteristics originally used to establish the optinum cesium reservoir temperature for maximum power was employed as a diagnostic tool.

The emitter temperature was held at T_E =1845°C throughout the test and the collector and cesium reservoir conditions varied as follows:

Test 1 - T_C =750°C, T_{Cs} =353, 363, 385, 408°C
Test 2 - T_C =730°C, T_{Cs} =383, 419°C
Test 3 - T_C =730°C, T_{Cs} =382, 383, 417
Test 4 - T_C =700°C, T_{Cs} =350, 374, 400, 430°C

The timing of these tests are as follows:

Between Test 1 and 2 - 20 minutes
Between Test 2 and 3 - overnight with T_C = 700°C
Between Test 3 and 4 - 2 hours

The results of the tests shown on Figure 3. The four curves shown for Test 1 exhibit the normal converter performance response to changes in cesium reservoir temperature. Specifically, the I-V characteristic indicates a low cesium reservoir temperature condition at T_{Cs} =353 and 363°C. At T_{Cs} - 385°C maximum power is produced (optimum cesium temperature) and an over optimum cesium reservoir temperature condition exists At T_{Cs} =408°C.

The I-V curve for the remaining tests, however, deviate in a manner which suggests a second cesium reservoir within the converter.

The dashed line (Test 2) is identical for T_{Cs} =383°C and 419°C, and is nearly identical to the I-V characteristic for T_C =750°C, T_{Cs} =385°C. The shaded band (Test 3) at T_C = 730°C indicates the range in I-V characteristic for values of cesium reservoir temperatures 382, 383, and 417°C. The

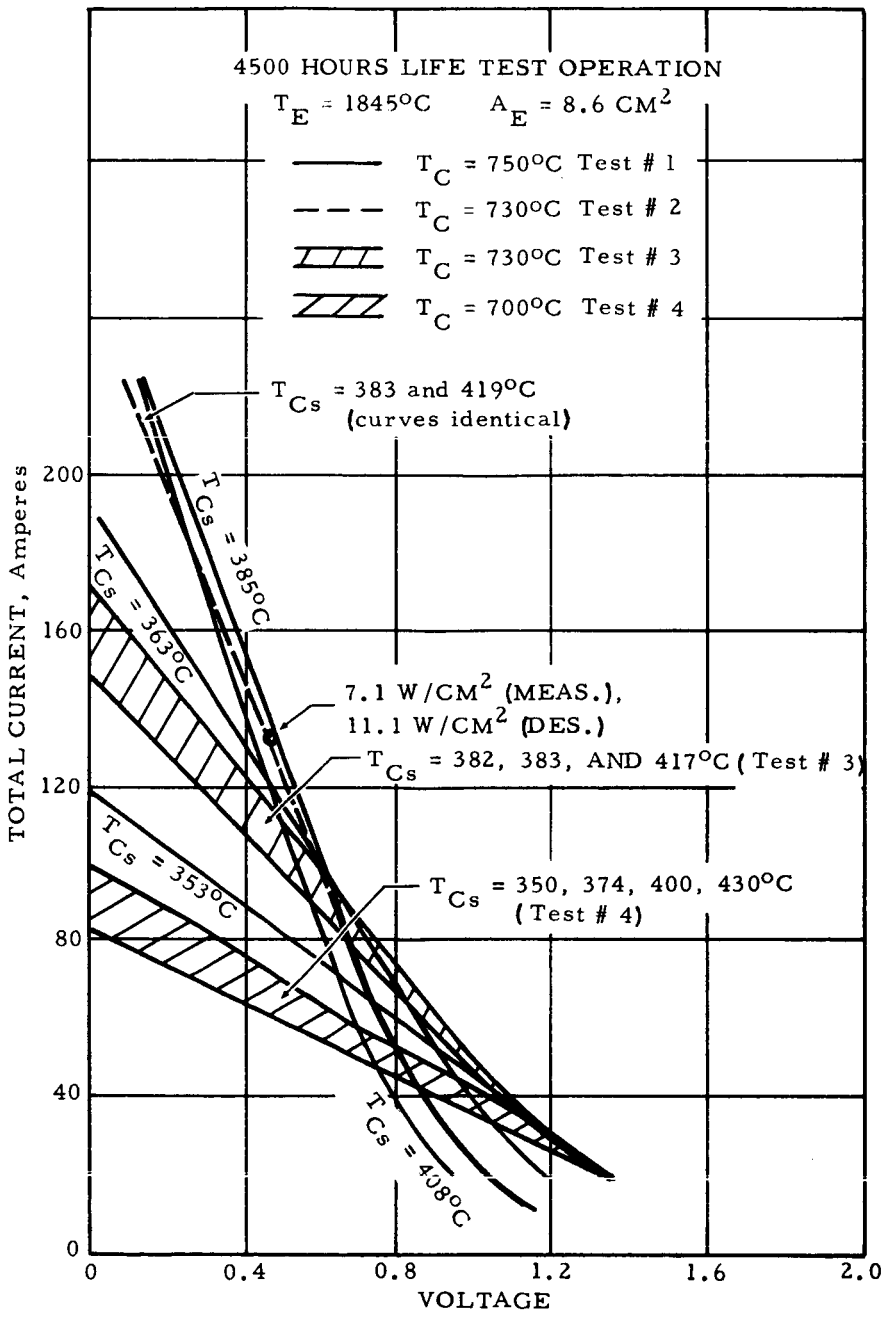


Figure 3 Converter 312 I-V Curves at Variable T_C and T_{Cs}

shaded band (Test 4) at T_C =700°C similarly indicates the range in I-V characteristic for cesium reservoir temperature values of 350, 374, 400, and 430°C. As can be seen in all cases where the collector temperature was below 750°C and after some elapsed time, the I-V characteristic curve at higher indicated cesium reservoir temperatures really exhibits a low cesium reservoir temperature condition.

The secondary reservoir seems to be primarily under the influence of the collector temperature and exhibits a time dependence. As tabulated on page 18, about 20 minutes elapsed between Tests 1 and 2; between Test 2 and 3 the collector temperature was held at 700°C overnight then returned to 730°C immediately before Test 3. A considerable difference can be seen in the I-V characteristic for two T_C =730°C tests (Tests 2 and 3).

The time difference between Tests 3 and 4 was 2 hours and a considerable change in characteristics is evident. The measurements made are not comprehensive enough to separate the effects of collector temperature from those of time. It is evident that the performance level of the converter has not been impaired.

Converter 313 - At the end of the reporting period, converter 313 had accumulated a total of 3775 hours and had completed six thermal cycles successfully. Input and output power for converter 313 plotted as functions of time are shown in Figure 4. The times at which the temperature cycles were impressed on the converter are also shown on this figure. The power output at the end of each of the cycles returned to its original value for the same input power and remains at this value between cycles.

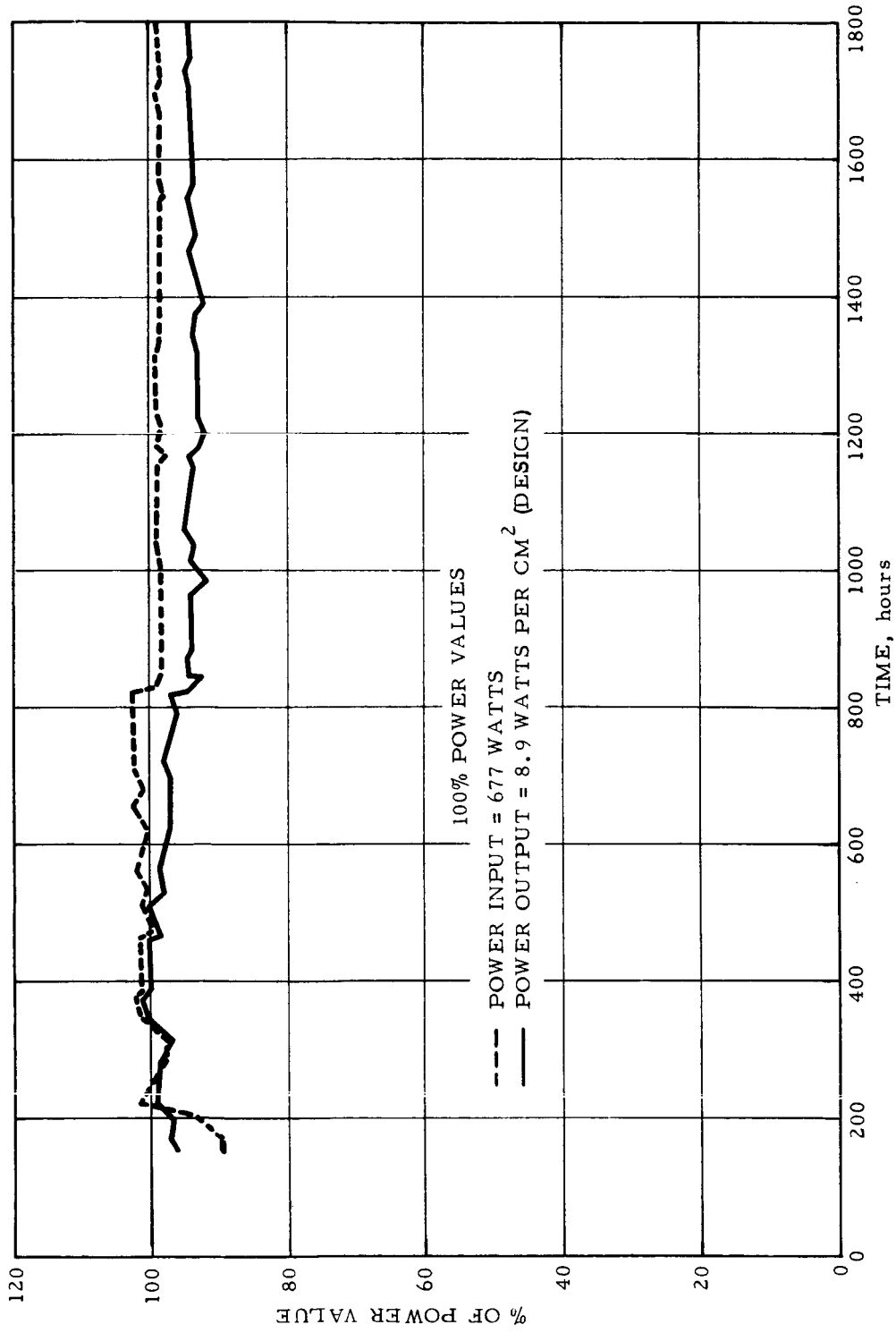


FIGURE 4a CONVERTER 313 POWER INPUT AND OUTPUT vs TIME

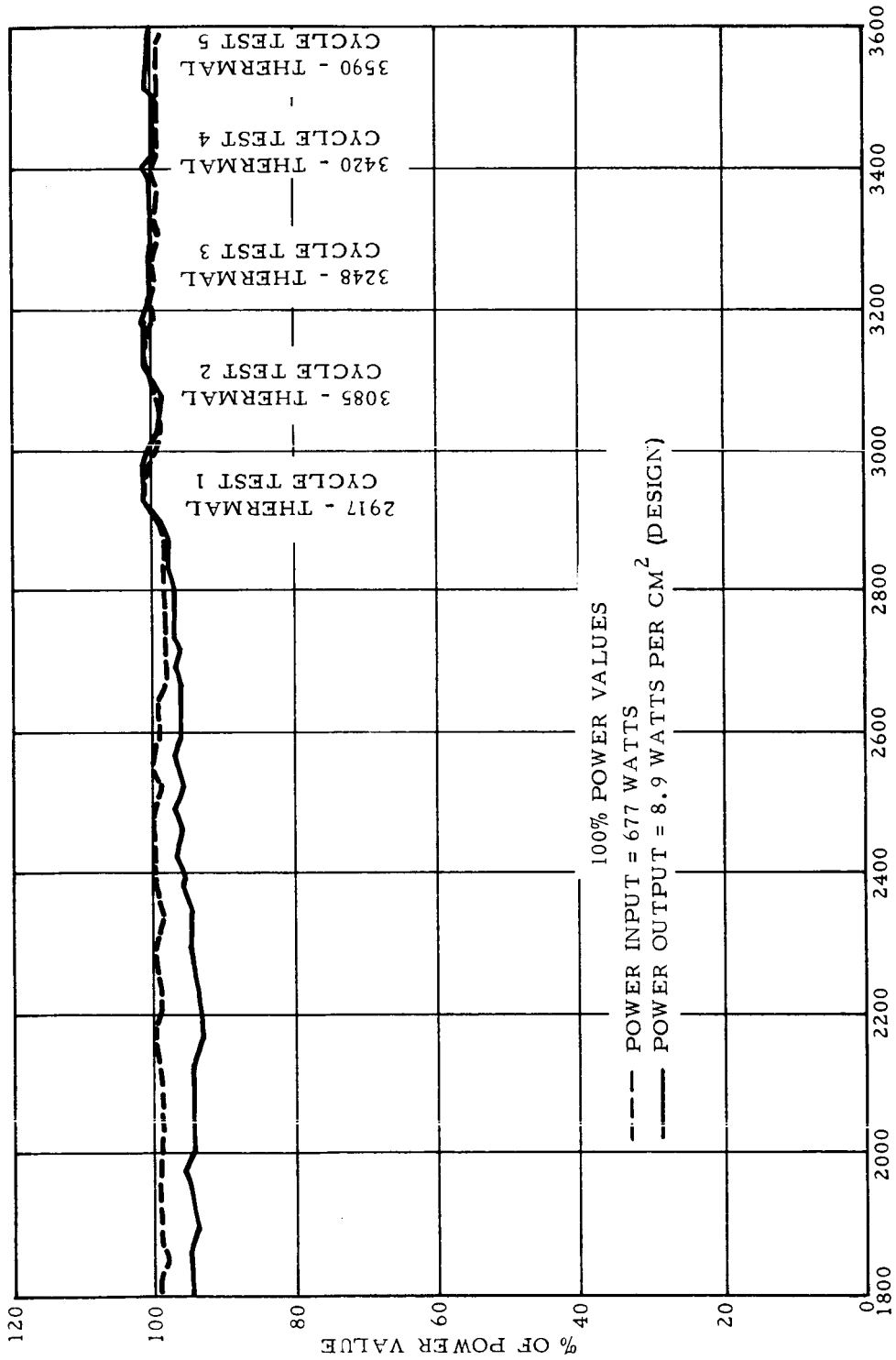


Figure 4b Converter 313 POWER INPUT AND OUTPUT vs TIME

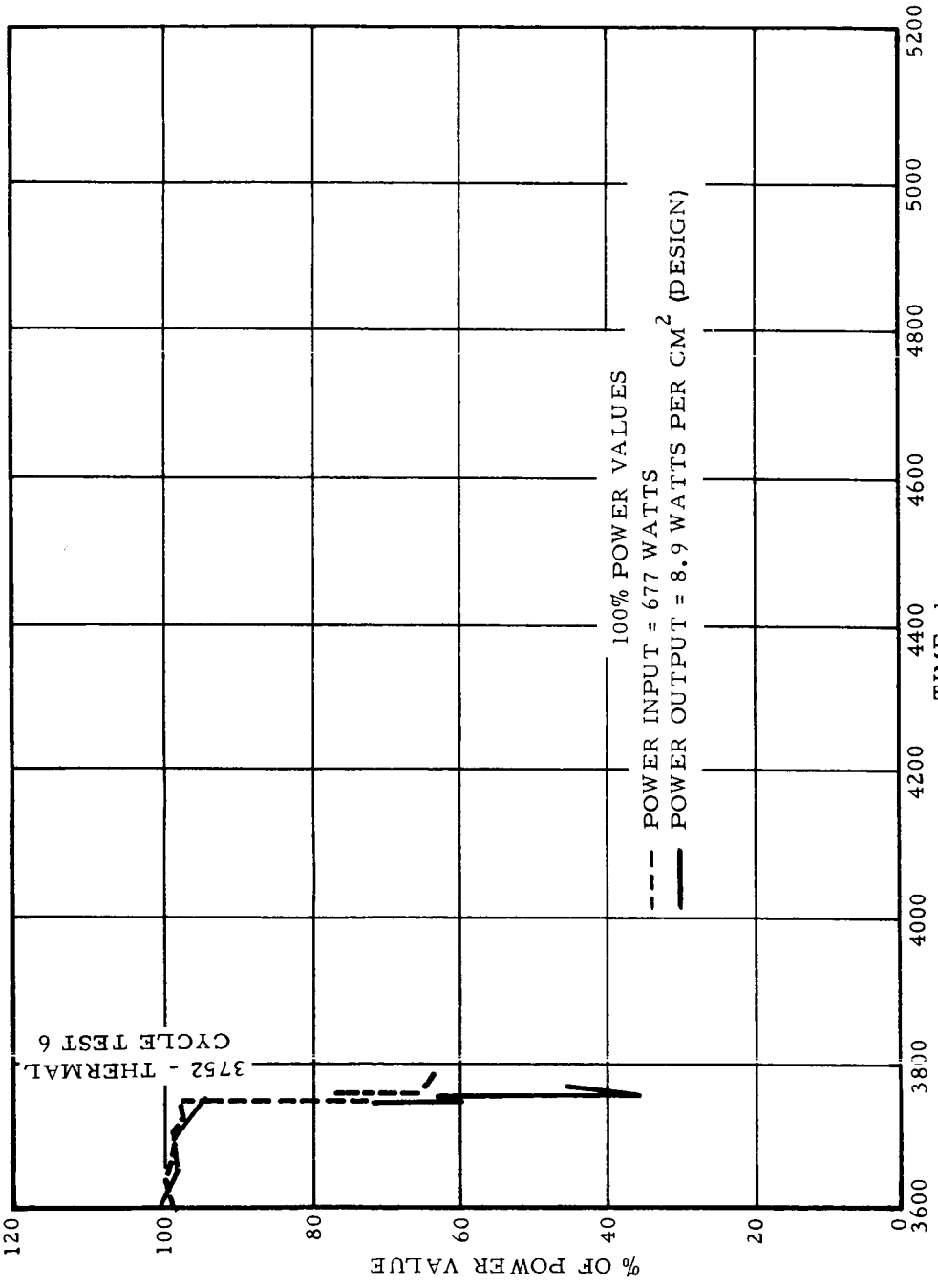


Figure 4c Converter 313 Power Input and Output vs Time

The temperature cycle that is impressed upon the converter is described as follows. Once each week the power in the emitter temperature is reduced to an emitter temperature of about 1400°C, the collector temperature to about 320°C. This point is held for about one-half hour at which time the converter is returned to its original input power value and an emitter temperature of 1830°C.. Converter performance is then observed at steady state power for one week at which time another temperature cycle is initiated. Shortly after Cycle No. 6, filament and bombardment characteristics indicated evidence of cesium in the bombardment chamber and the converter testing was terminated at 3775 hours.

The results of the converter life test through the seventh quarter are summarized in Table 1. The calculated thermionic design values for power output and efficiency were calculated using the same methods in the Second Quarterly Report.[1]

4. Task 3 and 4, Converters - Series 331 - 332

a. Layout drawings and assembly sequence of a three converter series-connected segment of a thermionic fuel element (TFE) based upon the thermal and stress analyses reported in reference 4 were submitted to the NASA Project Manager for his approval. Approval was obtained and the preliminary design of the out-of-pile converters (331-332) was initiated.

The out-of-pile design will reflect as closely as possible the design of the TFE. As many standard components as possible are being used.

TABLE I

COMPARISON OF CONVERTER CHARACTERISTICS

Conv. No.	T_E °C	T_C °C	P_{in} Watts	Elec. Spac. Inches	P_{meas} watts/cm^2	Emitter Surface Area/cm^2	V Meas. volts	I Meas. amps	P_{design} watts/cm^2	η_{meas} %	η_{design} %	Time on Test Hours
301	1830	680	402	0.010	7.4	8.6	0.695	91	9.1	9.4	12.9	3754
302	1830	700	386	0.010	7.9	8.6	0.555	122	11.3	8.2	12.6	100
303	1830	735	413	0.010	6.9	8.6	0.575	103	9.4	8.3	12.4	916
311*	1830	700	380	0.010	7.1	8.6	0.710	86	8.8	9.2	12.7	6413
312	1830	700	370	0.010	6.5	8.6	0.612	91	8.4	8.4	12.2	5807
313	1831	701	375	0.010	6.9	8.6	0.629	94	8.9	8.7	12.5	3775

T_E = emitter temperature

T_C = collector temperature

T_{Cs} = cesium reservoir temperature

P_{in} = total emitter power input

P_{meas} = measured power output

V_{meas} = measured output voltage at terminals on tube envelope

I_{meas} = measured output current

P_{design} = design power output (with no lead losses)

η_{meas} = measured efficiency

η_{design} = design efficiency

*311. For purposes of plotting P in and P out as a function of time the 100% P o value was taken at 780 hours when the load resistance was changed to retain emitter temperature. The value of V measured was 0.52 V. I measured was 90.5 amps. P measured was 5.5 watts/cm^2 and P design was 7.4 $\frac{\text{watts}}{\text{cm}^2}$

b. The mockup of the intercell connectors between the emitter and the adjacent collector has continued in test. During this quarter twenty additional thermal cycles were run bringing the total to sixty. After removal from test no indication of distortion or failure was observed except for one local region on the end disc of the collector. In this region, approximately 60° displaced from a lead pin, there was what appeared to be a grain boundary crack. This "crack" is shown in Figure 5. It is located near the heat affected zone of the weld joining the cylindrical and disc portions of the collector. Since it is displaced from the leads it is not believed to be associated with strains from the lead pin.

During cycling, the collector is cooled by a molybdenum heat sink that fits tightly into the collector on the cylindrical surface, but does not contact the end disc. Heat from the emitter is conducted through the lead pins into the cylindrical portion of the collector and into the heat sink. The end disc of the collector or receives energy from the emitter by radiation. Since the collector is at a comparatively low temperature, $1000^{\circ}C$, it does not radiate appreciably to the heat sink, but must reject its heat past the lead pins. As a result, the center of the end disc operates at a higher temperature than the edges. This was visually observed during cycling. At these temperatures the niobium upsets but also moves the cylindrical surface radially outward. During the cooling down, the cylindrical surface restrains the disc contraction and creates a radial, tensile stress in the end disc which causes partial yielding. Upon continued cycling a crack could develop. The development of this crack is not a significant event regarding these tests since, in an operating converter, the collector need not be leak tight and the structural and electrical functions of the end disc would not have been impaired.

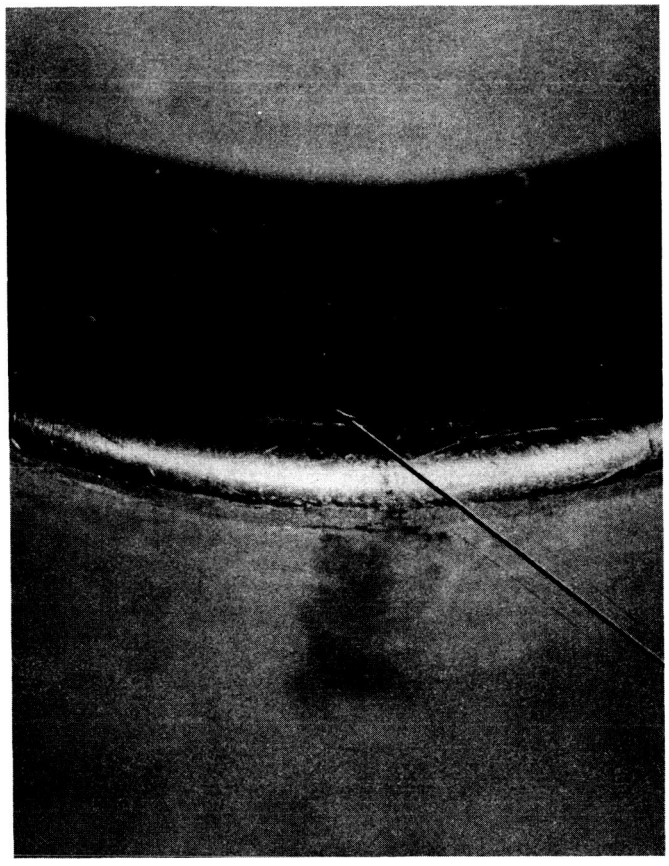


Figure 5 Photograph of the Suspected Crack in the Collector End Disk of a Mockup Cycled Sixty Times

5. Task 5, Converters - Series 431 - 433

These converters will be basically the same as the series 301-303 converters with the added provision for longitudinal slots to contain the UO_2 within the emitter. The design of the collector has been modified to allow operation at 1000°C for 4500 hours as the life objective. The design of the converters was shown in Figure 11 of reference 4.

During the reporting period the metallographic and spectrographic analyses of the tantalum thimbles has allowed a selection of satisfactory thimbles for inclusion in the three converters. The results of this analysis and the analysis of the braze joints on the collectors has been resolved and design approval has been received subject to the satisfactory documentation of the tantalum thimble inspection and the collector braze joint analysis. This documentation has been partially completed during this reporting period. Receipt of design approval has permitted the initiation of final assembly of the converters.

Two electron bombardment filaments were calibrated and one of these was used in the assembly of the first emitter for the temperature calibration of the emitter. The emitter calibration assembly is shown in Figure 6. During the first series of temperature runs, the glass viewing port in the vacuum system cracked and the pressure increased to greater than 300 microns. The emitter was at 1600°C and all components oxidized irreparably. New assembly and fabrication was continuing at the end of the report period.

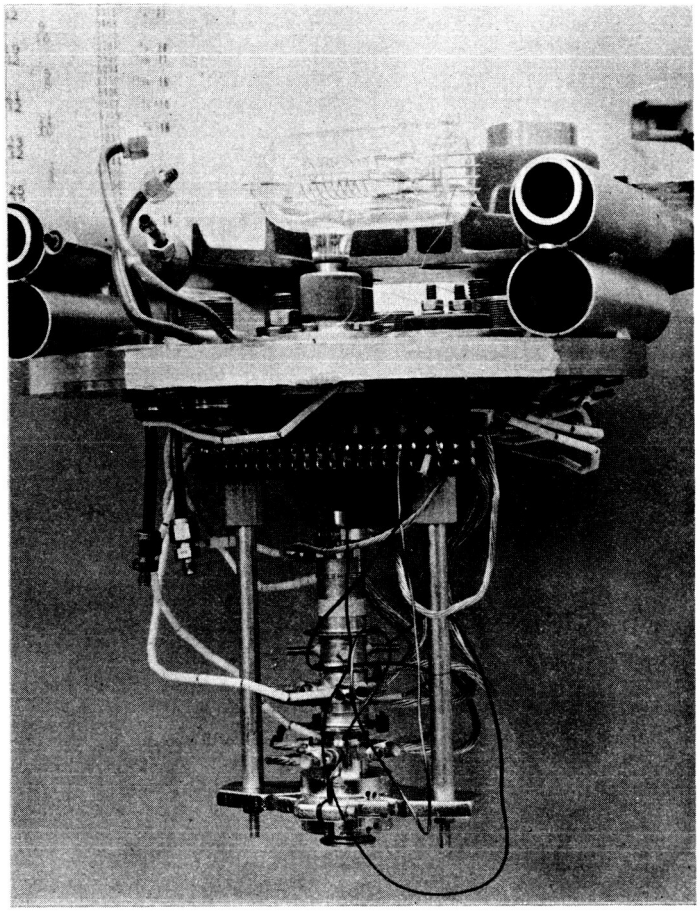


Figure 6 Emitter Temperature Calibration Setup

B. THERMAL AND IRRADIATION TESTING OF FUEL/CLAD EMITTERS

1. Long Term High Temperature Compatibility Tests of UO_2 Clad with W-25% Re

a. Summary

The objective of this task is to evaluate the long term chemical compatibility of UO_2 with W-25% Re, at elevated temperatures. Capsules of UO_2 clad with W-25% Re are being tested isothermally in gaseous cesium (1 to 5 torr) at 1800, 2000, and 2200°C for times to 5000 hours. Details of the test program and the progress of tests at the end of this report period are given in Table II.

Capsules 3, 4, 11 and 12 were examined after thermal treatment. No unusual changes were found in the dimensions of any of the capsules or in the weights of capsules 3 or 4. Capsules 11 and 12, however, showed a considerable weight loss because of rupture of the cesium containment chamber during the first 500 hours of testing. All four capsules have been sectioned and examined metallographically. No gross interaction between the UO_2 and W-25% Re was observed in any of these capsules. Traces of interaction were found, however, in capsules 11 and 12 only (2200°C). Discontinuous grain growth in the W-25 % Re was observed in capsules 11 and 12 (2200°C). Some evidence of porosity was observed in capsule 4 (1800°C) and capsules 11 and 12 (2200°C). Observations on each of the four capsules are presented in this section under Results and Discussion (III, B, 1, c). Experimental

TABLE II

PROGRESS OF FURNACE TESTS

Capsule[a] Number	Test Temperature °C	Target Test Time Hours	Time In Furnace Hours
1[a]	1800	2000	1950
2[b]	1800	2000	1950
3[c]	1800	500	Completed
4[c]	1800	1000	Completed
5	1800	3000	2197
6	1800	3000	2197
7	1800	5000	2197
8	1800	5000	2197
9	2000	2000	1038
10	2000	3000	1038
11	2200	500	Completed
12	2200	1000	Completed

a. All capsules had a 0.020-inch vent hole in one cap.

b. Cladding of all capsules except 1 and 2 was sintered W-25% Re from Chase Brass. The cladding of capsules 1 and 2 consisted of extruded tube from NMI and sintered caps from Hoskins.

c. All capsules except 3 and 4 were TIG welded. Capsules 3 and 4 were brazed with Mo-50% Re.

procedures, outlined in the previous quarterly,[4] are elaborated further and concluded in this report.

b. Experimental Procedures

Fabrication and Characterization of Capsule Components

The fabrication procedure and the chemical and metallographic analyses of the UO_2 pellets was reported previously.[4] Metallographic analysis of extruded NMI tubing (used in capsules 1 and 2) and chemical and metallographic analyses of the Hoskins end caps used with the NMI tubing were also discussed previously.[4] Chemical analysis of the NMI tube is given in Table III.

The W-25% Re used in capsules 3 through 12 was supplied by Chase Brass and Copper Company. The tubing and end caps were prepared by powder metallurgy techniques. Tubing was fabricated to a size considered optimum for machining by using the filled billet method. Final sizing was done by electrical discharge machining (E.D.M.), grinding and lapping. The required shapes of end caps were made by E.D.M. from hot rolled W-25% Re sheet. Longitudinal and transverse sections of the tube are shown in Figure 7. No evidence of sigma phase was detected at 500X.

Assembly of Capsules

Capsule assembly methods were described in the previous quarterly.[4] All capsules were assembled by TIG

TABLE III

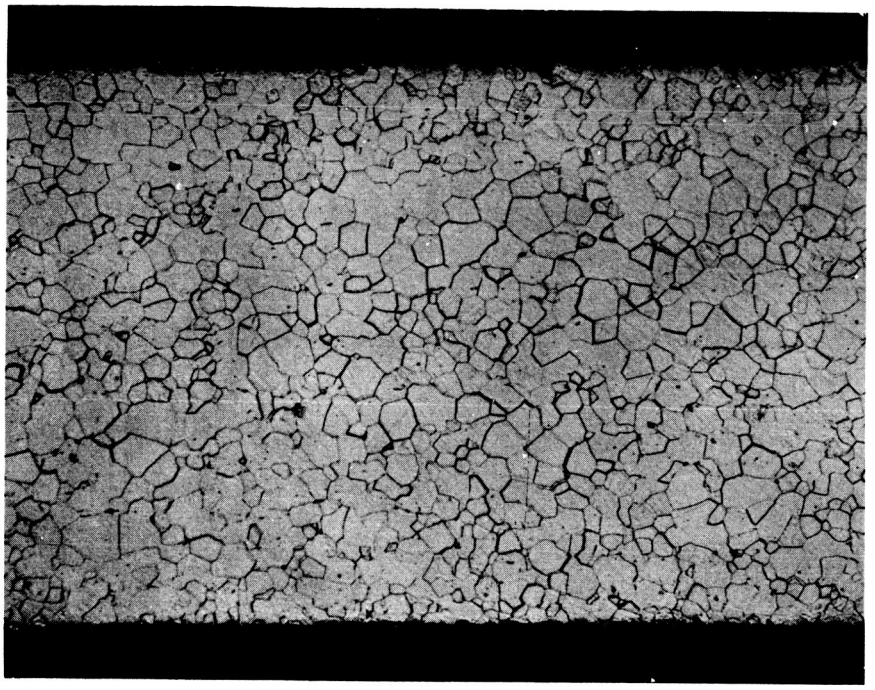
CHEMICAL ANALYSIS OF W-25% Re CLADDING

Element	NMI Tube	Chase Brass Caps	Chase Brass Tube	Analytical Techniques
Re	24.86%	24.89	24.95%	Gravimetric Precipitation
C	32 ppm	26 ppm	31 ppm	Conductometric
O	60	40	50	Inert Gas Fusion
N	120	100	100	Micro Kjeldahl
Al	< 10	< 10	10	Emission Spectrographic
Ca	< 1	< 1	< 1	Emission Spectrographic
Cr	< 10	< 10	< 10	Emission Spectrographic
Cd	< 20	< 20	< 20	Emission Spectrographic
Cb	< 50	< 50	< 50	Emission Spectrographic
Cu	19	20	18	Emission Spectrographic
Cs	< 50	< 50	< 50	Emission Spectrographic
Fe	11	7	35	Emission Spectrographic
Hf	< 100	< 100	< 100	Emission Spectrographic
Mo	182	115	125	Emission Spectrographic
Ni	8	5	10	Emission Spectrographic
Na	< 5	< 5	< 5	Emission Spectrographic

TABLE III

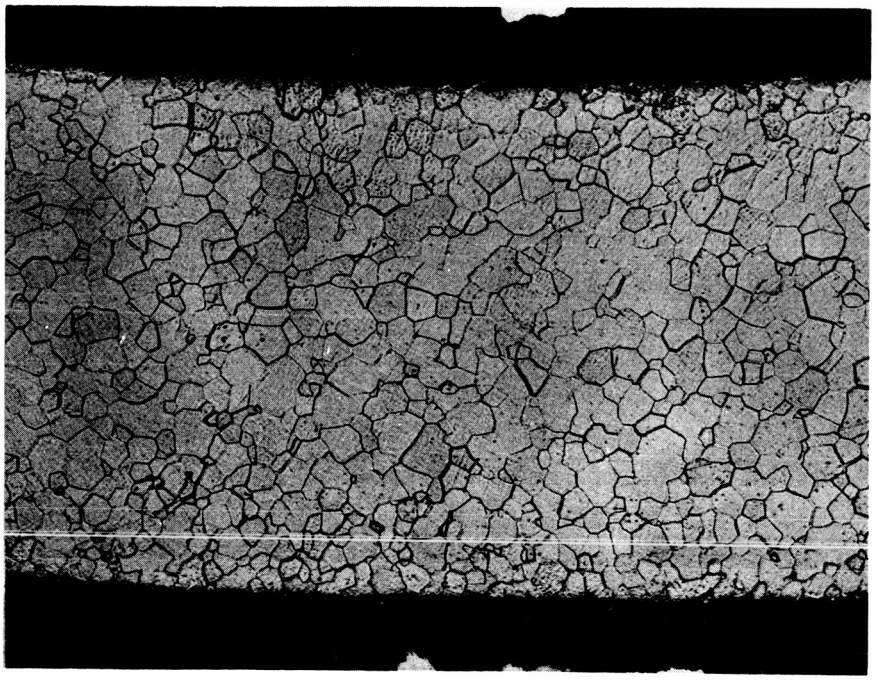
(Continued)

Element	NMI Tube	Chase Brass Caps	Chase Brass Tube	Analytical Techniques
Si	< 20	< 20	< 20	Emission Spectrographic
Sn	< 20	< 20	< 20	Emission Spectrographic
Ta	< 100	< 100	< 100	Emission Spectrographic
Ti	3	1	2	Emission Spectrographic
Zr	4	1	3	Emission Spectrographic
Zn	< 100	< 100	< 100	Emission Spectrographic



ETCHED 150X

7a. Longitudinal Section



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7b. Transverse Section

Figure 7 W-25% Re Tube from Chase Brass.

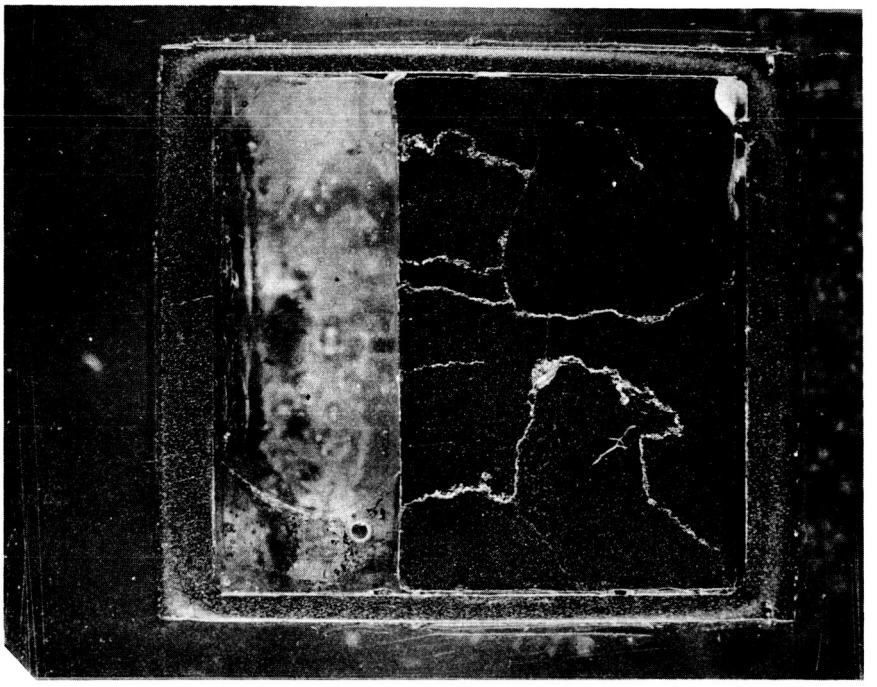
welding except capsules 3 and 4, which were brazed with Mo-50% Re. After assembly, each capsule was macroscopically examined and its weight and dimensions recorded. Photomicrographs of an as-brazed and an as-welded capsule are shown in Figures 8 and 9, respectively. An interface between UO_2 and the W-25% Re cladding, typical of both the as-brazed and as-welded capsules, is shown in Figure 10. The grain size and hardness of the W-25% Re cladding of each capsule are given in Table IV.

Metallographic Preparation

Each capsule was prepared for metallographic examination by: (1) vacuum filling it with epoxy to keep the UO_2 pellet in place in the capsule during sectioning and polishing operations; (2) sectioning it longitudinally and mounting the section in epoxy; and (3) applying routine grinding and polishing procedures. Structural details were revealed by etching the UO_2 (for 2 to 3 minutes) with a solution of 90% H_2O_2 and 10% H_2SO_4, and by etching the W-25% Re (30 to 40 seconds) with a solution of 1 part of 30% potassium ferricyanide and 1 part of 10% NaOH.

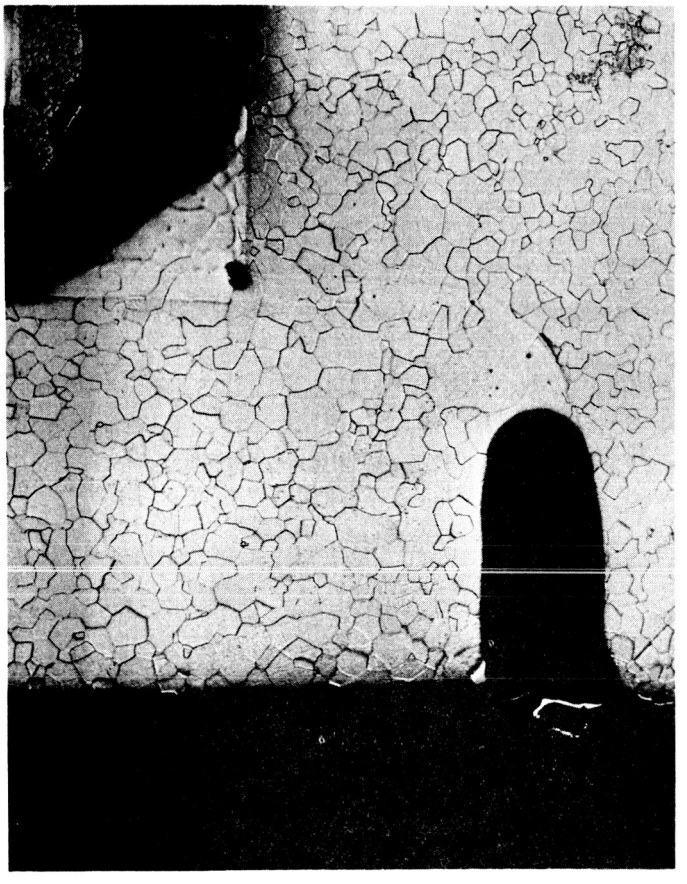
c. Results and Discussion

Thermal testing of capsules 3, 4, 11, and 12 was completed during this report period. External capsule surfaces were free of defects and showed no evidence of attack by cesium. Dimensional and weight changes



6X

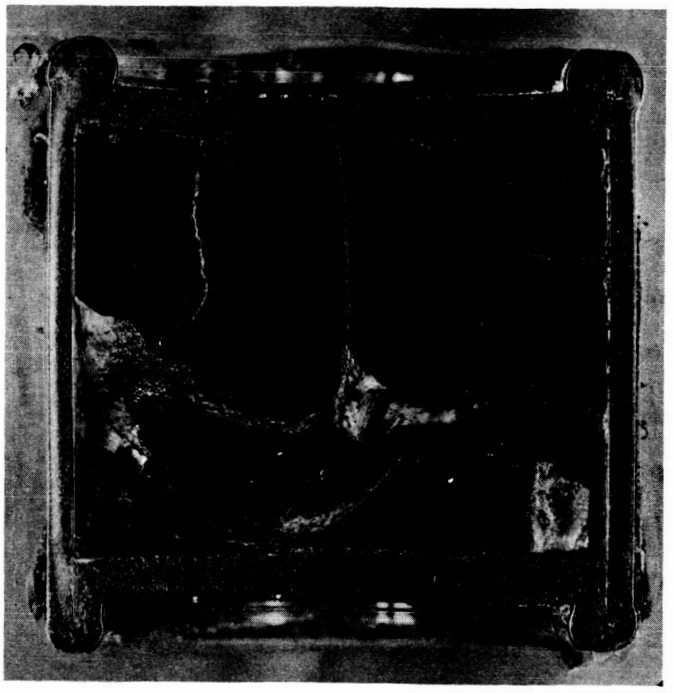
8a. Macroshot of Capsule



8b. Typical Braze Area

100X

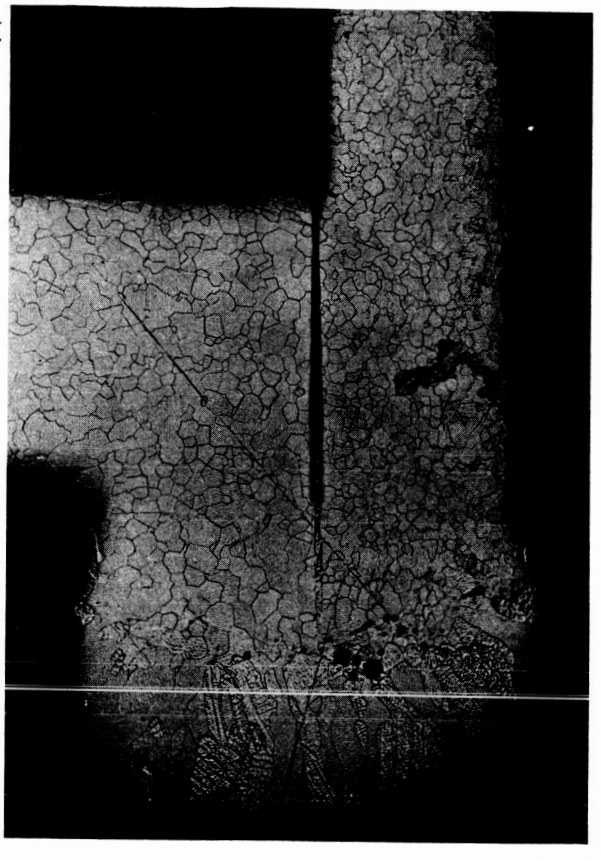
Figure 8 As-Brazed Capsule



9a. Macroshot of Capsule

6X

9b. Typical Weld Area



50X

Figure 9 As-Welded Capsule

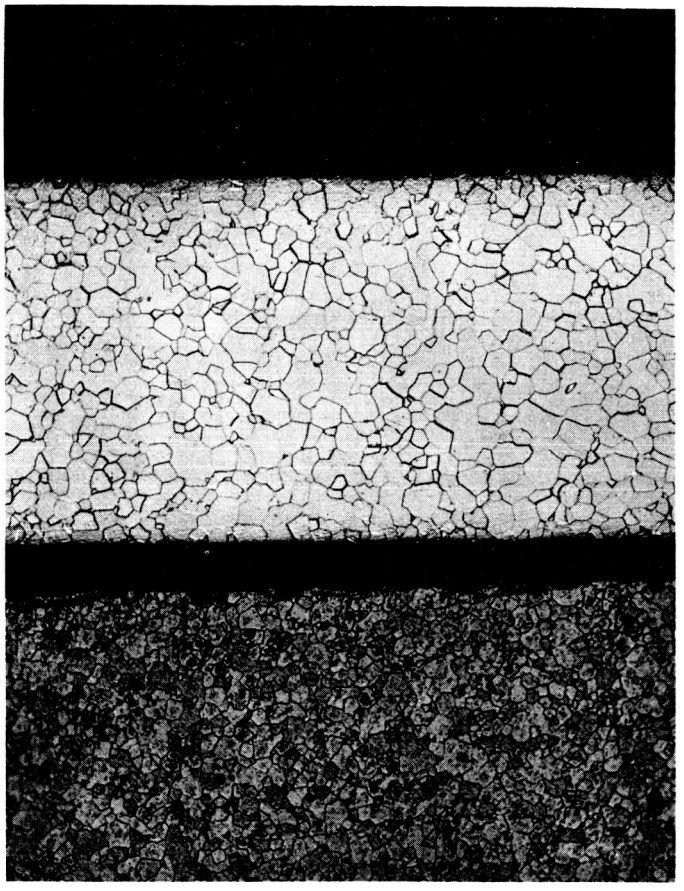


Figure 10 Typical Interface in As-Brazed and As-Welded Capsules.

TABLE IV

CAPSULE MEASUREMENTS

Capsule Number	Grain Size (ASTM) W-25% Re		Hardness (DPH) W-25% Re		Dimensional & Weight Changes		
	Wall	Cap	Wall	Cap	ΔD^a (in.)	ΔH^b (in.)	ΔW^c (gms.)
As-brazed	6.6	6.7	442	446	-	-	-
As-welded	6.6	6.7	453	449	-	-	-
3	6.0	7.0	455	511	None	None	0.0033
4	7.0	6.8	438	497	None	None	0.0052
11	Very large grains						
	5.2	n.d.[d]	420[e]	470[e]	None	None	2.1318
12	Very large grains						
	6	5.9	449[e]	450[e]	+0.002	None	2.0750[f]

a. ΔD = difference between pre- and post-test capsule diameter. Approximate initial diameter = 0.450 inch.

b. ΔH = difference between pre- and post-test capsule height. Approximate initial height = 0.450 inch.

c. ΔW = difference between pre- and post-test capsule weight. Initial weight = approximately 13 grams. Weight UO_2 = approximately 6 grams.

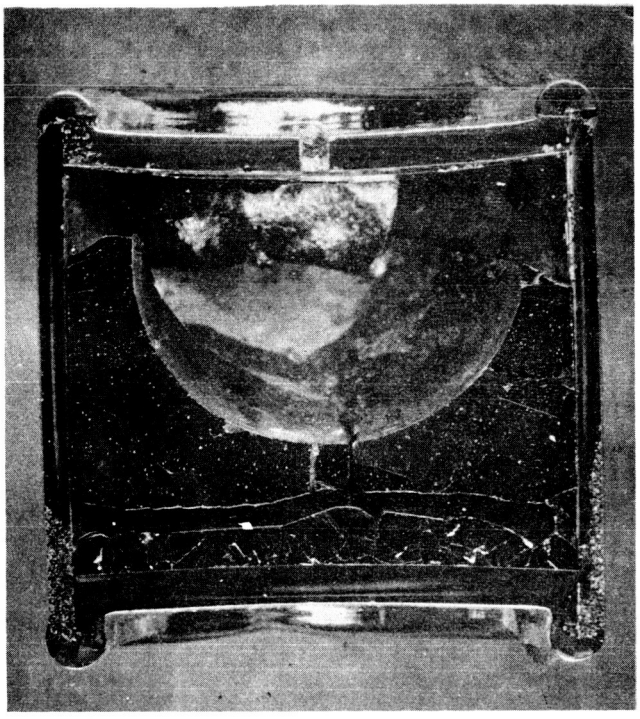
d. n.d. = no determination

e. Hardness readings refer only to areas of small equiaxed grams.

f. Weight loss during last 500 hours was 0.0064 grams.

of the capsules are recorded in Table IV. The large weight losses of capsules 11 and 12 were caused by the rupture of the tantalum chamber during the first 500 hours of the test. The rupture of the chamber permitted the escape of the Cs cover gas and exposed the capsules to vacuum in which greatly increased evaporation of UO_2 occurred. The weight loss of capsule 12 in the last 500 hours of thermal treatment was 0.0064 grams, which indicates the effectiveness of the cesium cover gas in suppressing evaporation. UO_2 was lost from each capsule as clearly shown in Figure 11. After inspection and measurements, the capsules were sectioned and examined metallographically (Figures 11 and 12). These capsules, like those examined during the first year of this program, exhibited no gross interaction between UO_2 and W-25% Re. Typical interfaces of each capsule are shown in Figure 13 and 14. No sigma phase was seen at 500X in any of the cladding. Two interesting effects were noted in the cladding of some of the capsules: abnormal grain growth and development of porosity.

The walls and caps of both of the 2200°C capsules showed very large grains running, sometimes, almost the entire length of a cap or wall. Much smaller equiaxed grains covered the residual areas. The grain growth is shown in Figure 12 and at larger magnification in Figures 14a and 15. Grain growth was normal in capsules 3 and 4. Grain sizes and hardnesses of



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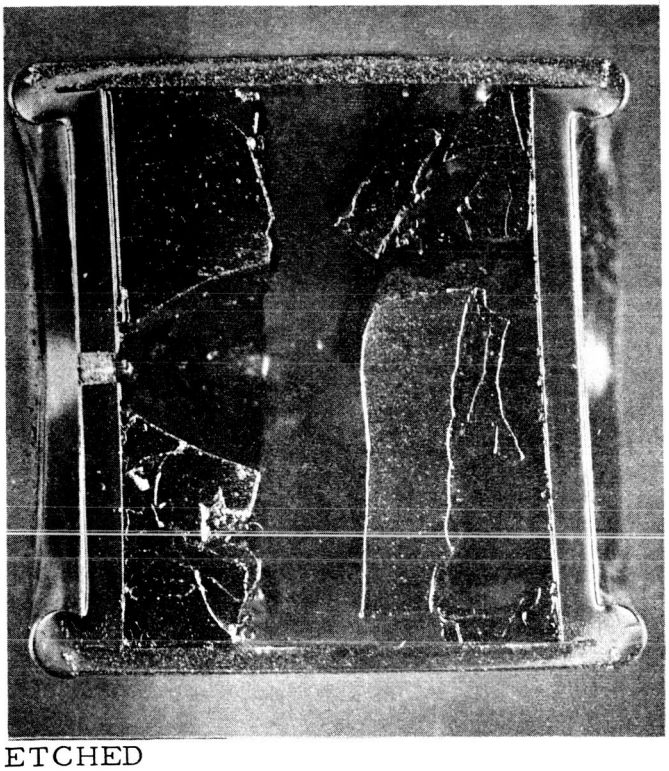
11a. Capsule 11 - 500 hours



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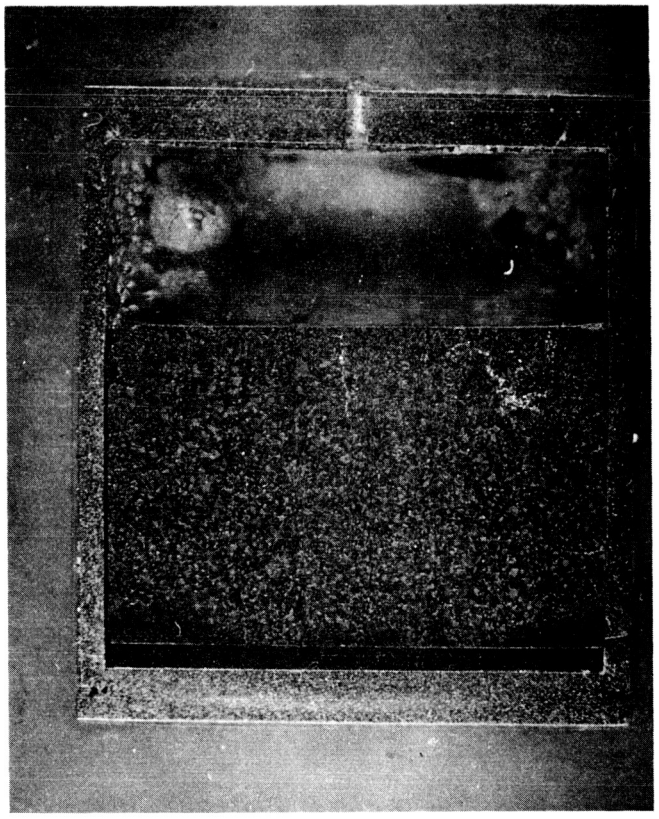
11b. Capsule 12 - 1000 hours

Figure 11 Macroshots of Capsules Tested at 2200°C.



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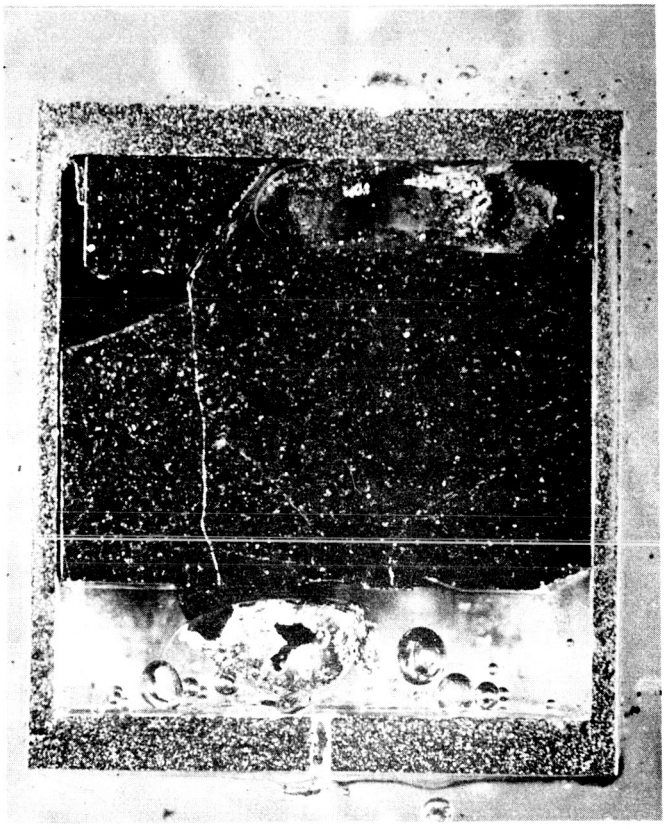
12a. Capsule 3
500 hours



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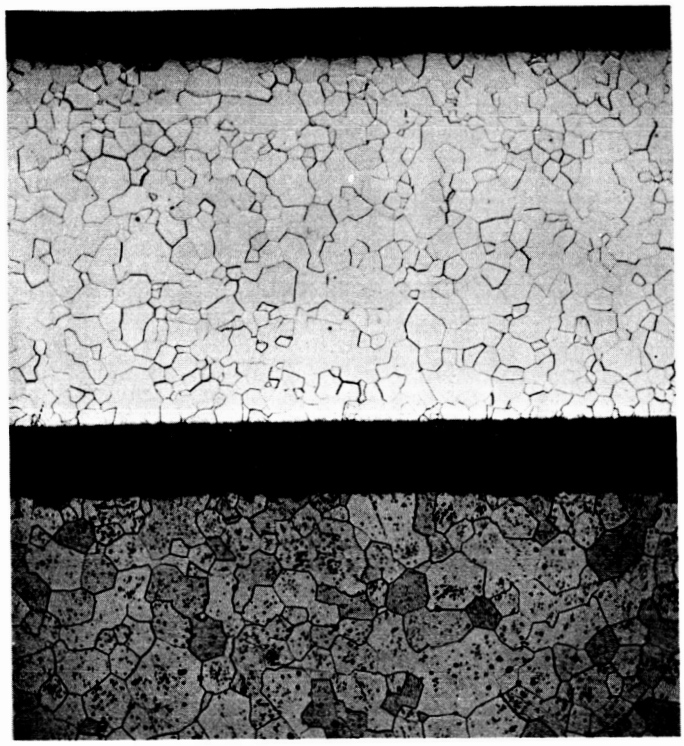
12b. Capsule 4
1000 hours

Figure 12 Macroshots of Capsules Tested at 1800°C



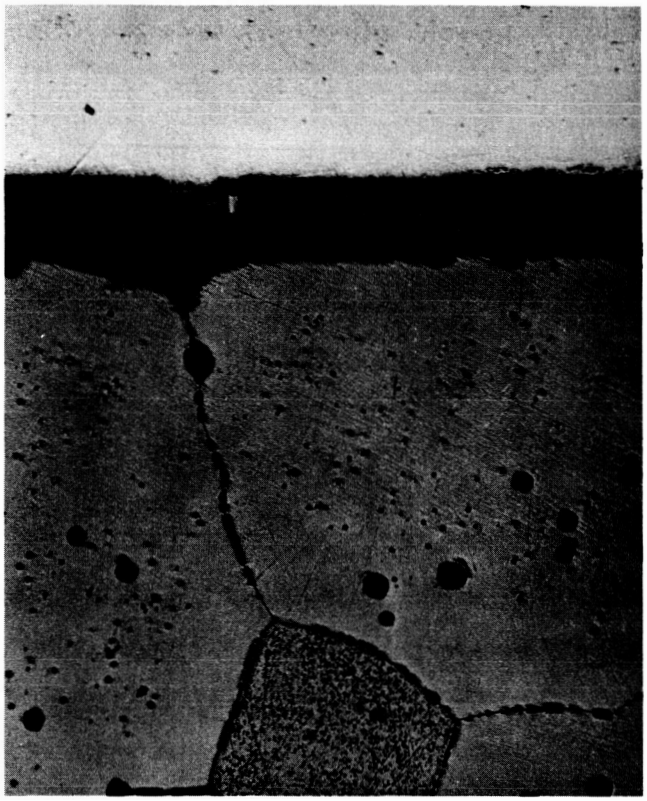
ETCHED 100X

13a. Capsule 3
500 hours

ETCHED 100X

13b. Capsule 4
1000 hours

Figure 13 Typical Interfaces of Capsules Tested at 1800°C

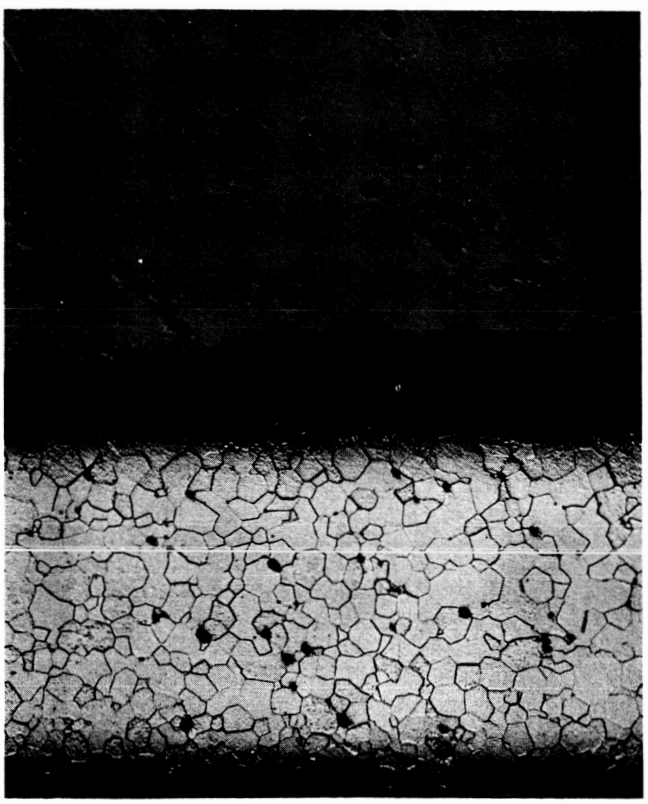


UO_2

14a. Capsule 11
500 hours

W-25% Re

ETCHED 100X



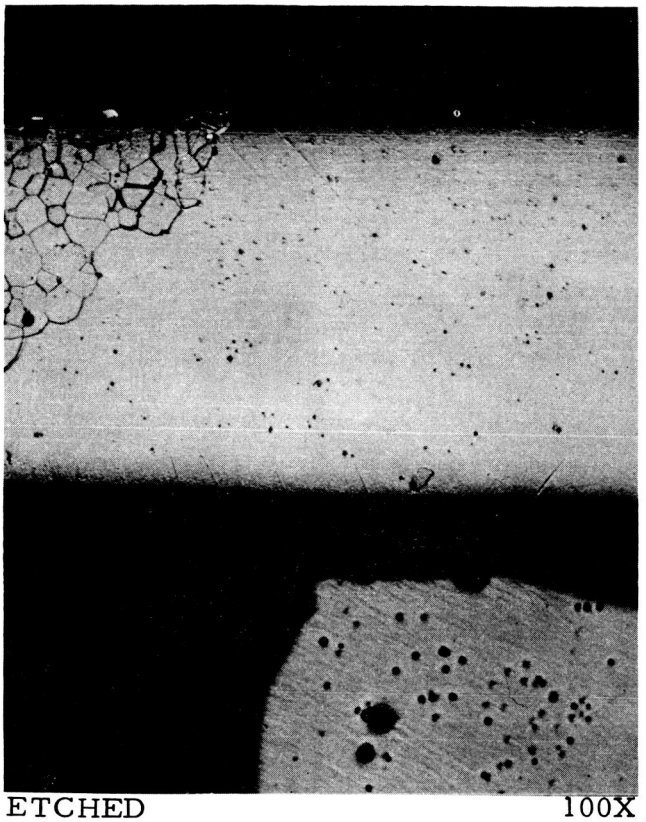
UO_2

14b. Capsule 12
1000 hours

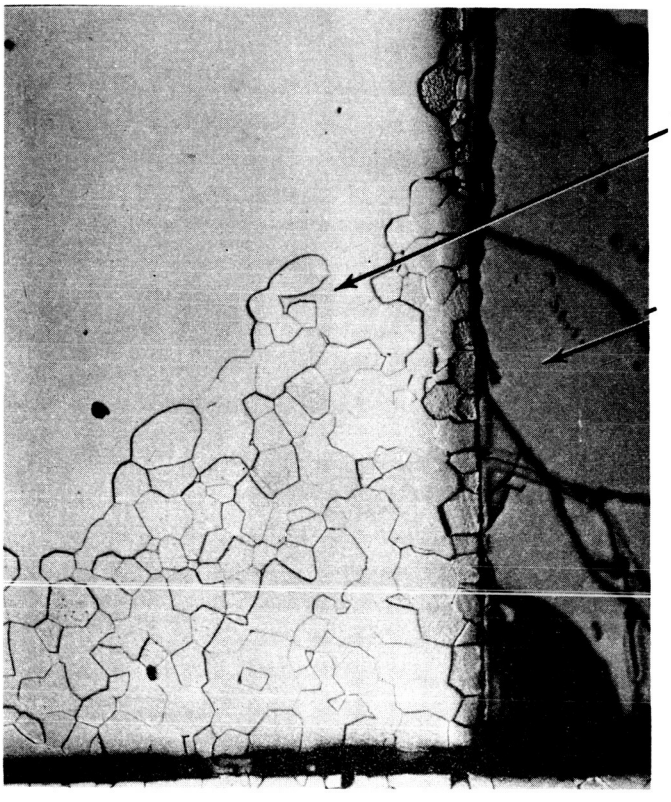
W-25% Re

ETCHED 100X

Figure 14 Typical Interfaces of Capsules Tested at 2200°C



15a. Wall of Capsule 11
500 hours



15b. Cap of Capsule 12
1000 hours

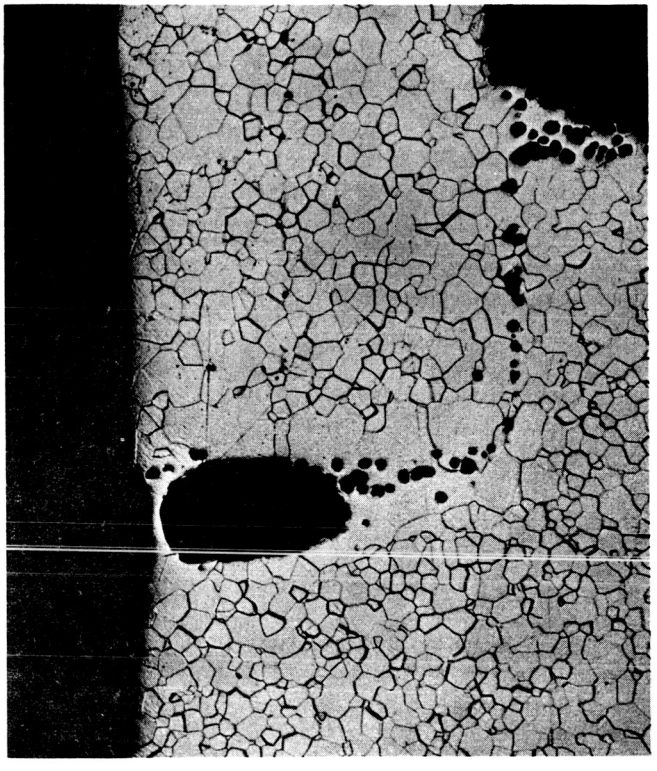
Figure 15 Discontinuous Grain Growth in Capsules Tested at 2200°C

the four capsules are tabulated in Table IV. Porosity was first noticed in the W-25% Re walls of capsule 4 (1800°C at 1000 hours). Compared to this capsule, the size and number of pores appeared to be greater in capsules 11 and 12 (2200°C). Very few pores were seen in the caps of these capsules, and in some samples none at all. Although metallography of as-received W-25% Re showed no evidence of pores, radiography indicated some light porosity in sections of the tube. It may be, therefore, that the pores observed in the thermally treated samples were associated with pores present in the tube before testing. Another explanation may be that micropores present, but not observable, at room temperature coalesced with increasing temperature and time. A third possibility is that the pores developed due to the accumulation and resulting pressure of impurities in the W-25% Re which could be gaseous at the high test temperatures.

Braze zones of capsules 3 and 4 are shown in Figure 16. Porosity was not present in the original braze area (Figure 8b), but began to develop along the center of the braze zone after 500 hours at 1800°C and was more evident after 1000 hours. The large void in Figure 16b similar to that shown in Figure 16a is due to incomplete filling in the braze joint and is not due to thermal treatment. The growth of pores is believed to be associated with the Kirkendall effect,[5] i.e., when two metals having different diffusion coefficients

ETCHED 100X

16a. Capsule 3
500 hours



ETCHED 100X

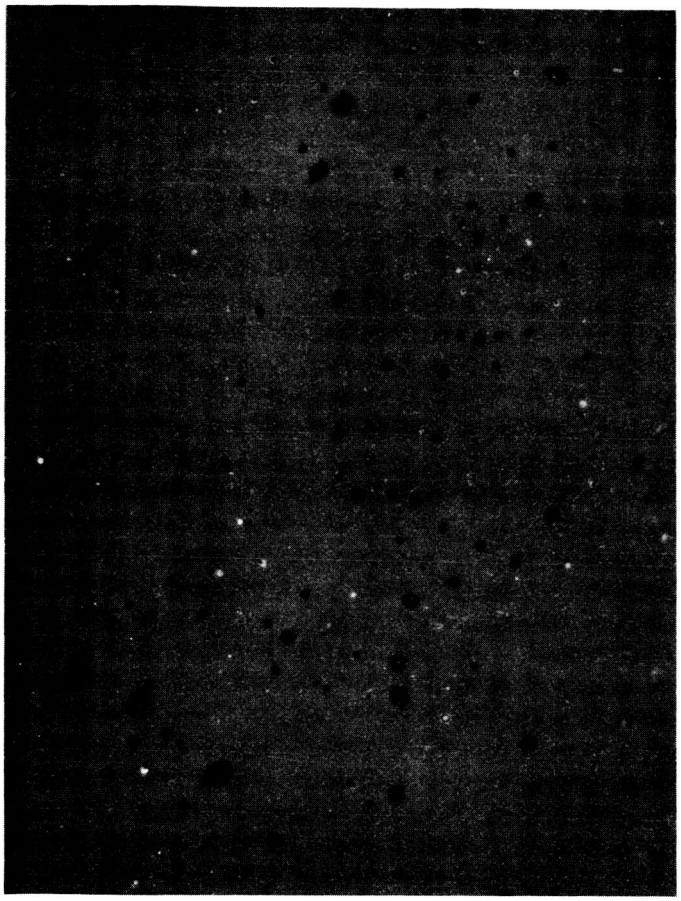
16b. Capsule 4
1000 hours

Figure 16 Braze Areas in Capsules Tested at 1800°C

are permitted to interdiffuse there is a net transport of material across the plane that initially separated the two. In the brazed samples, it is believed that molybdenum diffuses out of the braze area faster than tungsten can diffuse in the opposite direction, thus creating a void area in the braze.

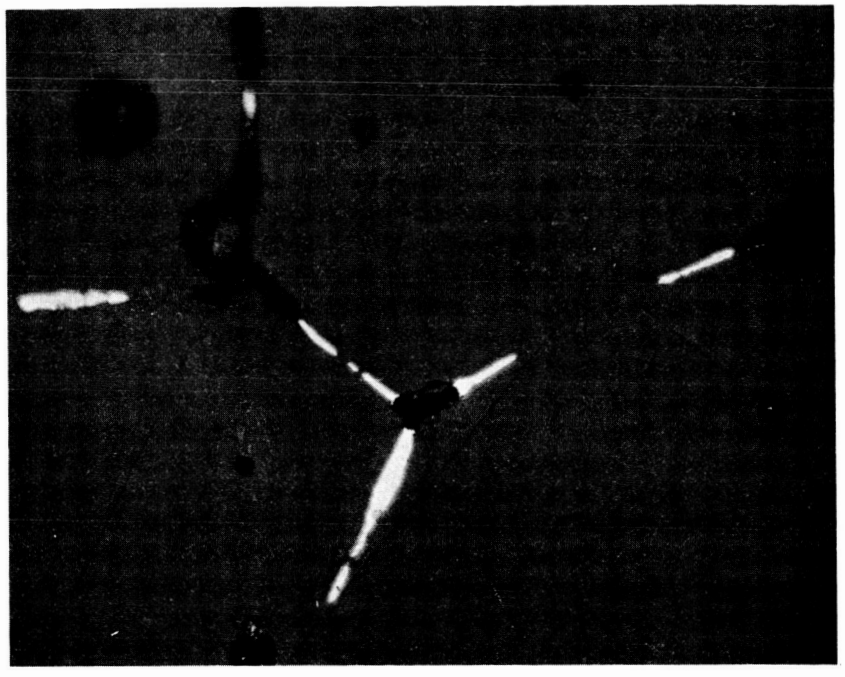
Hardness measurements were made across the braze areas. In moving from the center of the Mo-50% Re into the cladding the hardness gradually increased to a maximum value outside the original braze-W-25% Re interface and then decreased to the hardness of the cladding. The hardness maximum is probably associated with local zone of a hard ternary solid solution. Microprobe analysis would be required to evaluate concentration gradients in brazed areas to clearly establish the reason for braze porosity and the hardness peaks.

The UO_2 in all capsules contained metallic phase and, in addition, the UO_2 of capsules 3 and 4 contained a nonmetallic second phase. The metallic phase was present as randomly distributed very fine particles (Figure 17) in capsules 3 and 4. The metallic phase was precipitated in grain boundaries, pores and cracks in capsules 11 and 12 after thermal treatment at 2200°C, Figures 18 and 19 respectively. The amount of metallic phase seen in capsules 11 and 12 greatly exceeded the amount found in capsules 3 and 4. The metallic phase appears to be uranium, but microprobe analysis would



AS-POLISHED 1000X

Figure 17 Metallic Phase in Capsules 3 and 4



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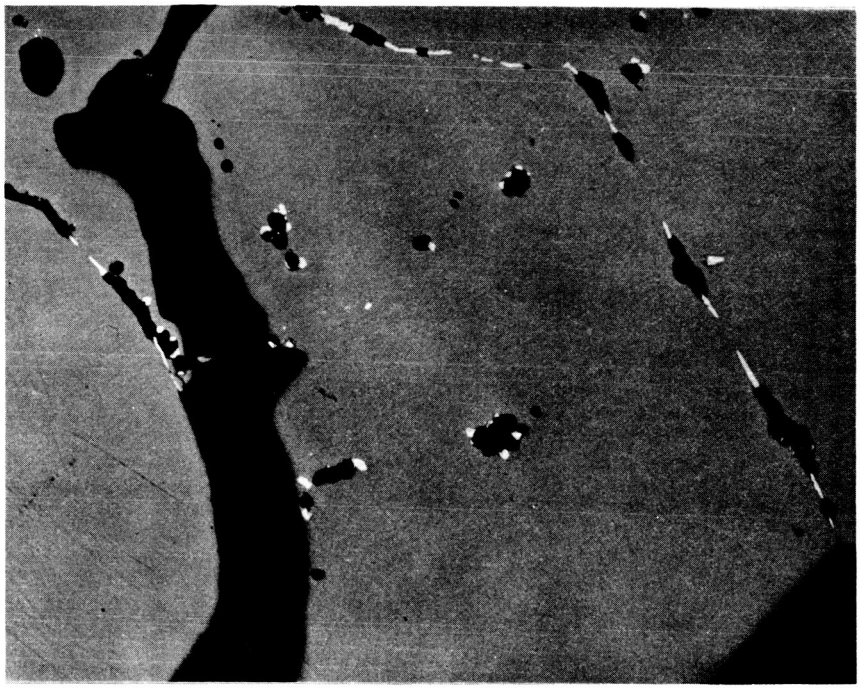
18b. Metallic Phase in UO_2 Pore

Figure 18 Metallic Phase in the UO_2 of Capsule 11 (2200°C - 500 hours)



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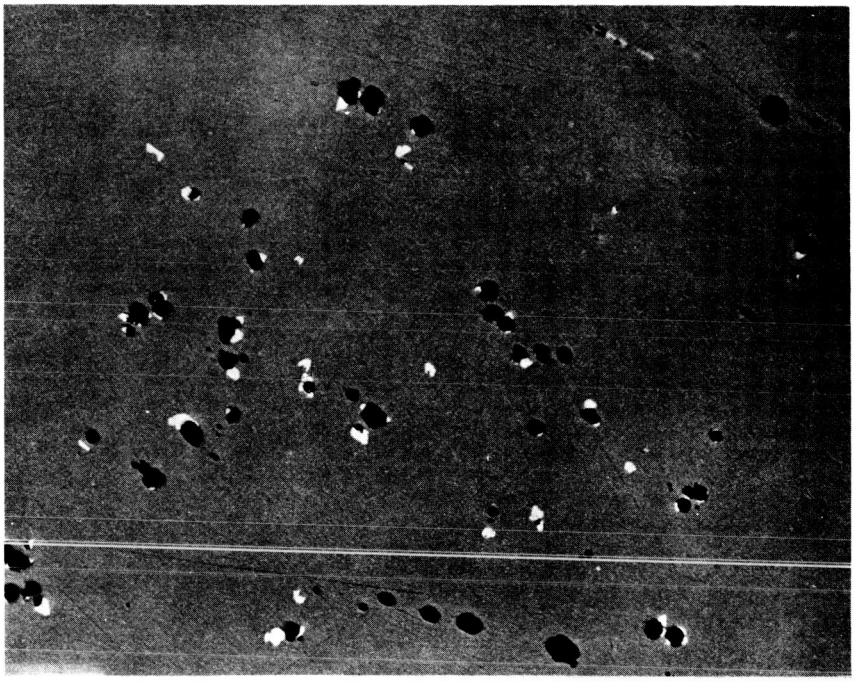
a.



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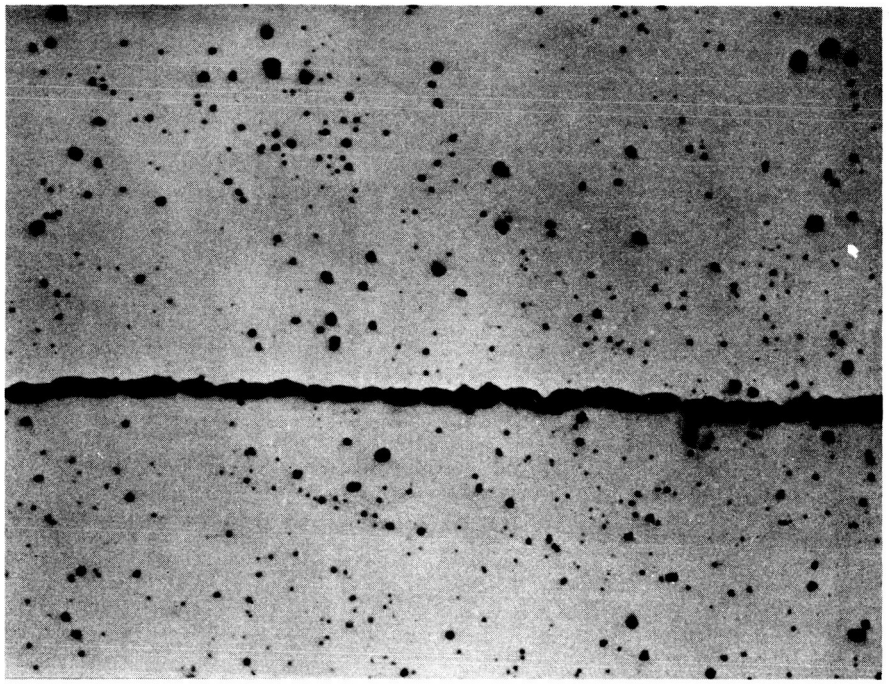
b.

Figure 19 Metallic Phase in Capsule 12 (2200°C - 1000 hours)

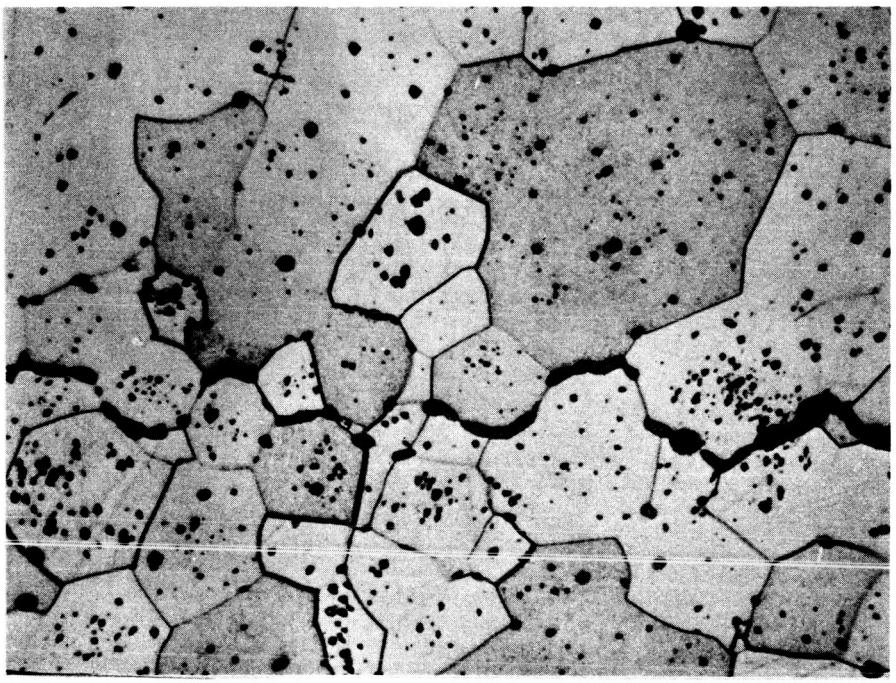
be necessary to make a conclusive identification. Uranium is expected in the UO_2 because of the high temperatures to which the samples were subjected. Experimental evidence[6-10] has shown that UO_2 becomes substoichiometric (UO_{2-x}) above 1600°C, releases oxygen, and retains the excess uranium in solution in the UO_{2-x} structure. The degree of substoichiometry increases with increasing temperature. Upon cooling, the UO_{2-x} reverts to the stoichiometric composition, $UO_{2.00}$, and ejects the excess uranium. Depending upon the rate of cooling, the uranium precipitates in varying amounts within the UO_2 grains and/or at grain boundaries and pores. The nonmetallic second phase observed in the 1800°C capsules (randomly distributed in as-fabricated UO_2 pellets[4]) was found to be concentrated in cracks, grain boundaries, and to a lesser extent, within grains, (Figure 20). The non-metallic phase was not seen in either of the capsules tested at 2200°C. The second phase may have evaporated at this high temperature. Microprobe analysis would be required to identify the nonmetallic phase.

Earlier, it was stated that no gross interaction between UO_2 and W-25% Re was formed in capsules 3, 4, 11 and 12. Traces of interaction were observed, however, in capsules 11 and 12. A thin layer (~ 1 micron thick) of what appeared to be metallic phase was found along the inner edge of the non-vented cladding cap, Figure 21.



AS-POLISHED 500X

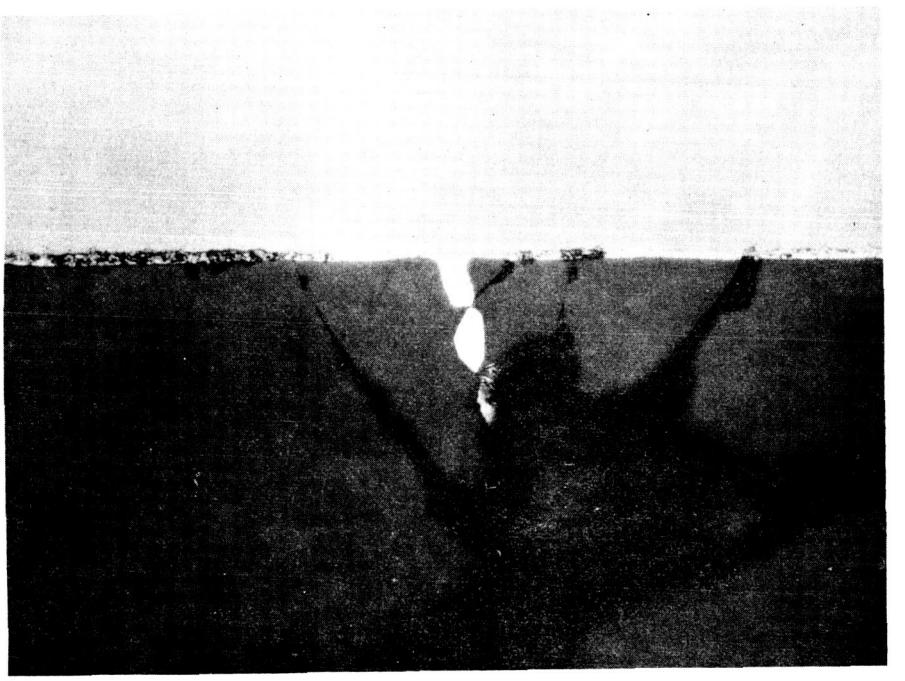
20a. Nonmetallic Phase Precipitated in Crack and Within Grains.



ETCHED 500X

20b. Nonmetallic Phase in Grain Boundaries and Within Grains

Figure 20 Nonmetallic Phase (Dark Grey) in UO_2 (Light Grey Background) of Capsules 3 and 4 (1800°C)



AS-POLISHED 1000X

Figure 21 Thin Layer of Metallic Appearing Phase Along the Interface Between UO_2 and Clad Cap Capsule 11

UO_2 etchant attacked the thin layer indicating the presence of uranium. The layer before and after etching is shown at very high magnification in Figure 22. Limited interaction was also seen at the interface of UO_2 and the vented cladding cap of capsule 12. A small amount of what appears to be UO_2 was found penetrating grain boundaries just inside the cladding, Figure 23 and 24. The interaction zone along the interface seems to be associated with what appears to be metallic phase in the UO_2. The metallic phase was attacked by UO_2 etchant. Microprobe analysis of the interaction zones would be required to identify the phase, but the small size of the zones would permit only qualitative answers at best.

2. Fuel/Clad Irradiation

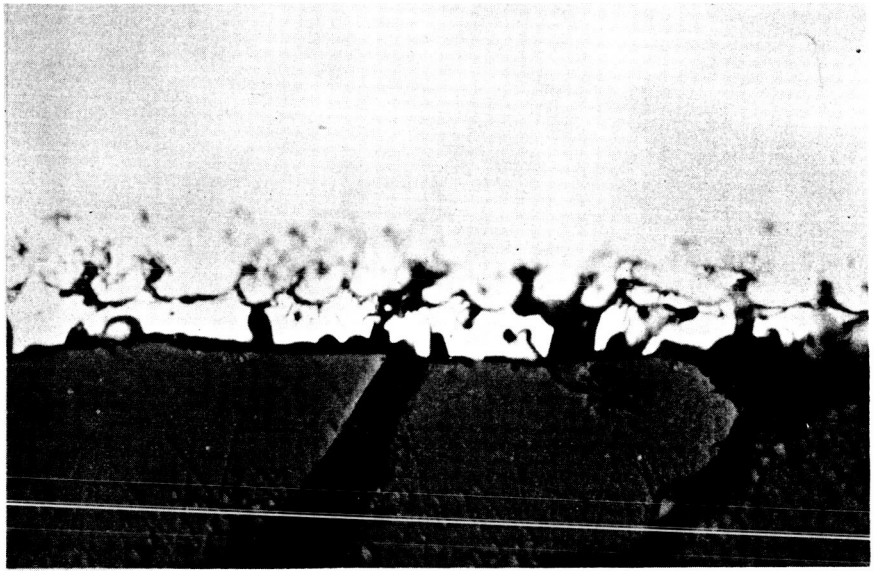
a. General

The objective of this task is to obtain data on chemical compatibility, dimensional stability, venting characteristics, weld microstructure, and fission product release for UO_2 fueled, W-26% Re clad emitters at temperature in nuclear environment. The task consists of irradiating specimens in the Plum Brook Reactor (PBR). Two irradiations are planned.

During the seventh quarter, redesign of the experiment was accomplished to attain a burnup in the UO_2 fuel of 1×10^{20} fission/cm^3 while minimizing irradiation time and utilizing as much of the fabricated capsule components

2000X

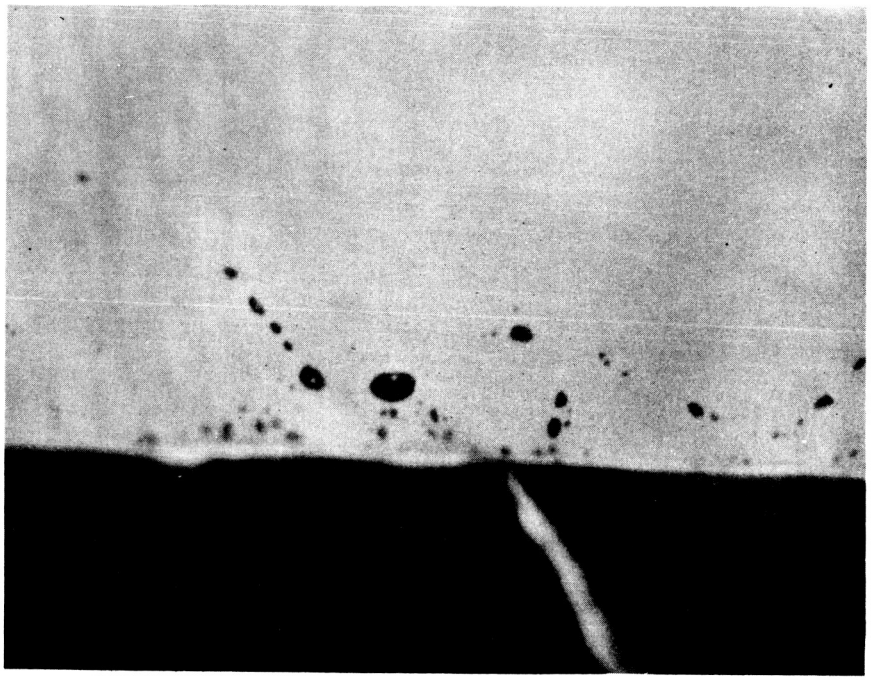
22a. As-polished



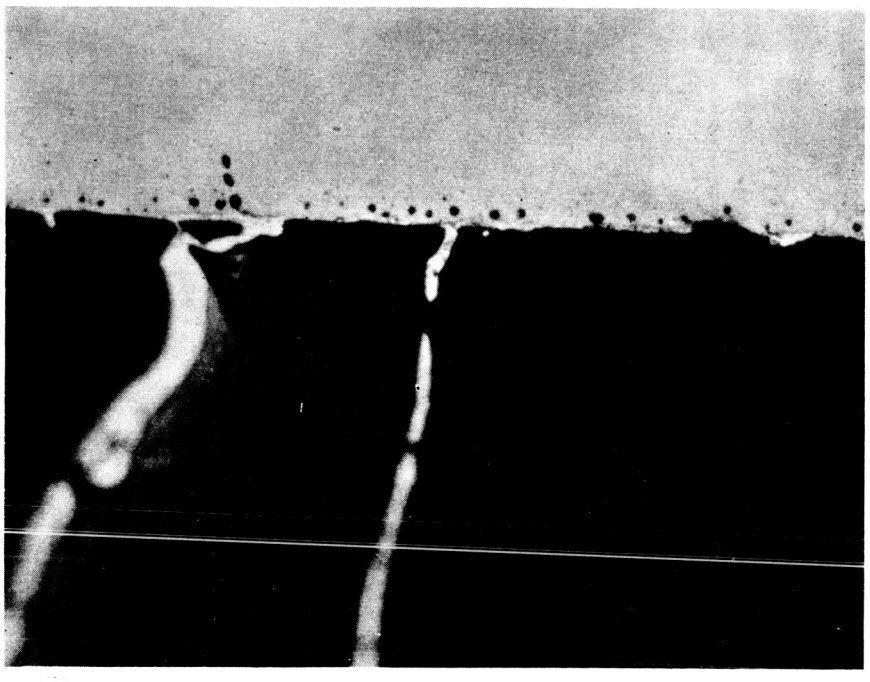
2000X

22b. Etched with UO_2 Etchant

Figure 22 Thin Layer of Metallic Appearing Phase Along the Interface Between UO_2 and Clad Cap Capsule 11



AS-POLISHED a. 1000X



AS-POLISHED b. 1000X

Figure 23 Nonmetallic Phase (Dark Phase) Penetrating W-25% Re Grain Boundaries Along Interface with UO_2. White phase in UO_2 Appears to be Metallic.

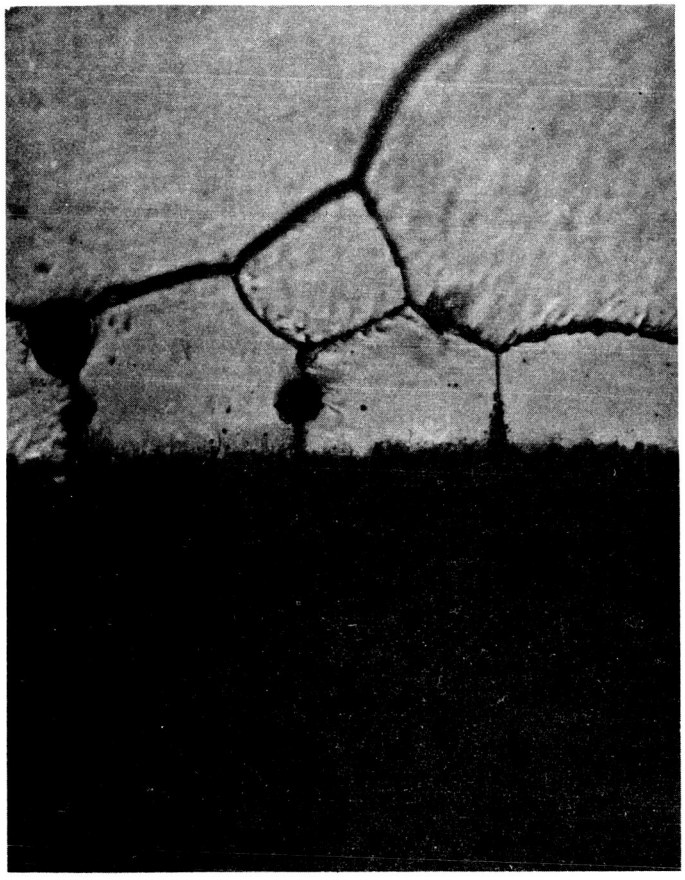


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Figure 24 Same General Area as Figure 23 Showing Nonmetallic Phase in W-25% Re Grain Boundaries

as possible. Modifications to the Design and Hazards Manual reflecting the redesign were accomplished. Investigations to permit definition of the test parameters for the second irradiation were initiated.

b. Objectives

The first irradiation of the 64-01 R1 experiment is designed to accomplish the following at a fuel burnup of 1×10^{20} fission/cm^3:

(1) Measure fission gas release from bulk UO_2 in an applicable thermal environment.

(2) Investigate the fission gas release mechanisms from peripheral cladding vents.

(3) Measure the fuel transport from peripheral vents in an applicable thermal environment with an inert cover gas.

(4) Investigate the compatibility of UO_2 with W-26% Re clad material at thermionic conditions in a nuclear environment.

(5) Determine the structural integrity of fueled emitter structures consisting of bulk UO_2 clad with 0.020 inch W-26% Re.

A summary of the pertinent test parameters is presented below:

Temperature-clad	1800°C + 0°C -100°C
Clad specimen environment	Approximately 75% argon - 25% helium gas mixture
Flux (thermal)	3 - 5 x 10^{14} n/cm^2/sec
Fuel burnup	Approximately 1 x 10^{20} fission/cm^3
Internal fuel temperature	<2300°C
Total emitter power	~650 watts
Surface heat flux - based on fuel	~64 W/cm^2
Test hole	PBR LD-11
Irradiation time	Approximately 2900 hours

The four specimens to be irradiated in the capsule are described as follows:

No.	Fuel	Clad	Vented	Location	Atmos. in clad	Enrichment
1	UO_2	W-26% Re (0.020)	3-0.020 dia. holes	Upper	1 atm 75% argon - 25% helium	2.4%
1	UO_2	W-26% Re (0.020)	3-0.020 dia. holes	Upper	1 atm 75% argon - 25% helium	2.4%
1	UO_2	W-26% Re (0.020)	3-0.020 dia. holes	Lower	1 atm 75% argon - 25% helium	1.5%
1	UO_2	W-26% Re (0.020)	3-0.020 dia. holes	Lower	1 atm 75% argon - 25% helium	1.5%

c. Design Analysis

The minimum time (2900 hours - approximately 8 calendar months in PBR) to achieve 1 x 10^{20} fission/cm^3 burnup was determined by establishing the maximum power allowable for the basic design based on PBR Technical Specification Change No. 15 requirements for capsule cooling. The containment can surface temperature for this test will not exceed the saturation temperature of the cooling water for maximum normal operating conditions at the maximum flux position for the capsule. Other transient conditions show the burnout factor is always in excess of 1.5 which meets the Technical Specification Change No. 15 requirements.

The increased heat flux from the specimens will be accommodated by utilizing a 75% argon - 25% helium gas mixture surrounding the specimens. Repositioning the capsule deeper into the PBR flux profile will permit the increased fission power.

Capsule Materials and Fabrication

As reported in previous quarterly reports, all of the materials are on hand. The quality control inspections have been completed satisfactorily and all material meets specification. The fabrication of a test specimen for hydrostatic test of a containment can is in progress.

The linear position indicator for the Vertical Adjustable Facility Tube (VAFT) has been received. The operational

checks and accuracy and repeatability tests on this unit have been completed with satisfactory results. The unit, shown in Figure 25, is ready for the installation of the VAFT drive unit and the control panel installation into the console.

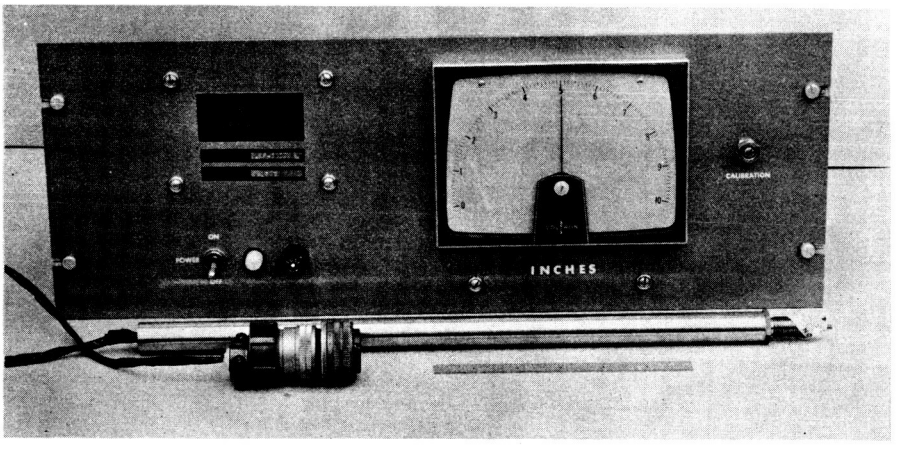


Figure 25 Linear Position Indicator Transducer and Control Panel

C. IRRADIATION OF ALUMINA

1. General

The capsule was irradiated in the Plum Brook Reactor for seven cycles during the period of 13 August through 21 December 1964. During this quarter the PBR Nuclear Analysis Section calculated the fast flux history of the experiment. An exposure of 1.8×10^{20} nvt greater than 1 MeV was reported. This is a 12.5% increase over that reported in the Sixth Quarterly Report where the exposure was estimated from the data log sheets. The nickel flux wire in the capsule will be counted as part of the post-operational examination. This will give a confirming value for the total exposure. The capsule was removed from PBR and the shell cut away in the PBR Hot Laboratory. The vacuum can was shipped to the General Electric Vallecitos Atomic Laboratory Radioactive Materials Laboratory where post-operational examination has been initiated.

2. Irradiation

An analysis was made to determine the fast and thermal flux history for cycles 23 to 29. The calculations were performed by the PBR Nuclear Analysis Section and the results appear in Table V.

The approach used to generate an average flux value for a cycle was to correct clean "168-gram" values for mixed loadings, flux tilting, regulating rod, and shim rod effects. The cycle average was defined as the arithmetic mean of the

TABLE V

SUMMARY OF FLUX HISTORY FOR EXPERIMENT 63-10*

Cycle	MWD	$\bar{\Phi}$ fast $\bar{\Phi} > 1$ MeV	$\bar{\Phi}$ fast/$\bar{\Phi}_{th}$	Fast neutron dose (>1 MeV) nvt	Uncertainty at 95% Confidence	Position
23	448.7	0.64×10^{14}	0.20	0.63×10^{20}	± 75%	LD-11
24	314.2	0.41×10^{14}	0.27	0.18×10^{20}	± 50%	LA-11
25	53.4	0.34×10^{14}	0.27	0.026×10^{20}	± 20%	LA-11
26	636.1	0.40×10^{14}	0.18	0.37×10^{20}	± 35%	LA-11
27	253.8	0.42×10^{14}	0.13	0.19×10^{20}	± 20%	LA-11
28	512.5	0.34×10^{14}	0.27	0.25×10^{20}	± 20%	LA-11
29	505.0	0.34×10^{14}	0.27	0.25×10^{20}	± 20%	LA-11
Total	2723.7			1.89×10^{20}		

*Data provided by PBR Nuclear Analysis Section

beginning and end of cycle values which were first vertically averaged over the experiment capsule region from + 2.7 inches to -4.3 inches.

The values shown in Table V incorporate all flux perturbations due to the experiment and loading configurations except those due to the experiment itself. The larger uncertainly for cycle 23 was due to the No. 5 shim rod effect which was difficult to evaluate accurately. Since the capsule flux perturbation factor with respect to fast flux are very nearly unity, the values in Table V are well within the limits of accuracy of the flux measurements and will be compared to flux wire data for further confirmation.

3. Data Analysis

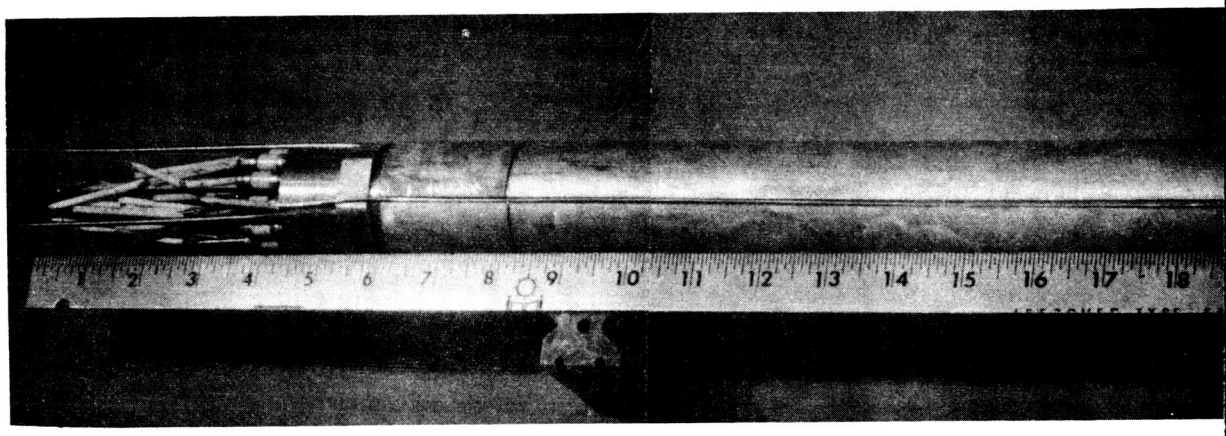
No additional work was done this quarter on Data Analysis. Results to date are presented in the Sixth Quarterly Report.

4. Post-Operational Examination

The capsule was removed from the Plum Brook Reactor during the shutdown following cycle 29. This was during the week of December 21, 1964. The capsule was cut from the lead hose assembly while still in the pressure vessel. The capsule was removed from the pressure vessel through the fuel transfer chute. For the cooling period the capsule was stored in the PBR fuel storage canal. On February 11 and 12 the capsule was disassembled in the PBR Hot Laboratory. The vacuum can was separated from the capsule

shell using the PBR Hot Lab remote hack saw and tubing cutter. No gross defect or damage was observed.

The vacuum can was packed and shipped to the Vallecitos Atomic Laboratory in a Series 200 cask. It was received March 12, 1965 and placed in a hot cell for examination. Shown in Figure 26 is the vacuum can as-received. Post-operational examination is currently in progress.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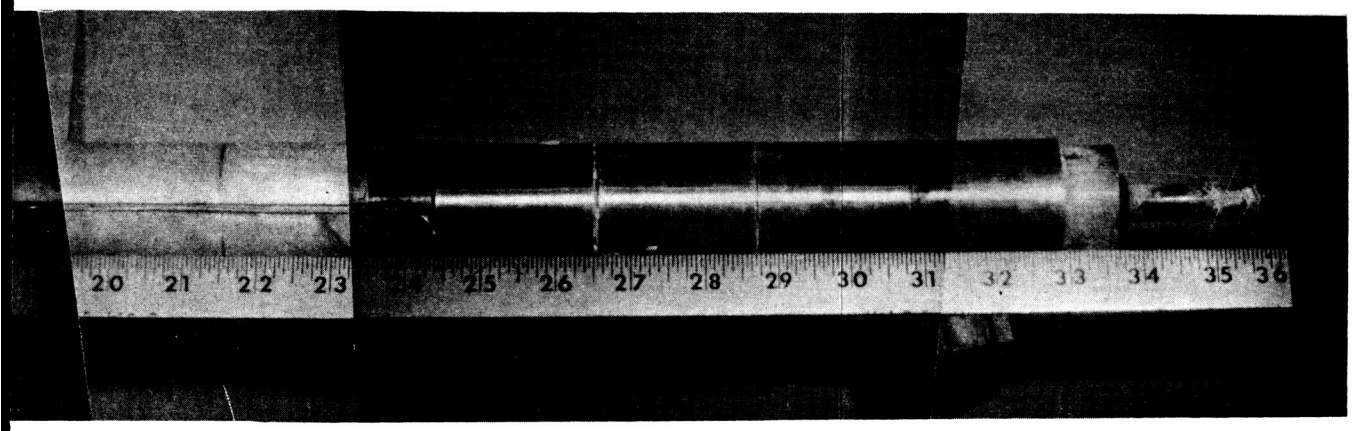

Figure 26

Insulator Irradiation Vacuum Can As-Received at Vallecitos Atomic Laboratory RML for Post-Operational Examination

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